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PROBLEMS ENCOUNTERED DURING INSTALLATION AND OPERATION  
OF A  
STORABLE PROPELLANT FACILITY  
FOR TESTING OF TITAN II COMPONENTS AND SYSTEMS

WYLE LABORATORIES

EL SEGUNDO, CALIFORNIA

TECHNICAL NOTE

10235

CONTRACT NO. AFO4(647)-398

MARCH 7, 1961

PREPARED FOR

AIR FORCE BALLISTIC MISSILE DIVISION  
AIR RESEARCH AND DEVELOPMENT COMMAND  
UNITED STATES AIR FORCE

AIR FORCE UNIT POST OFFICE  
LOS ANGELES 45, CALIFORNIA

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### FOREWORD

This Technical Note is prepared in accordance with Test Directive Number 28 of Air Force Contract Number AF04(647)-398, and is intended for preliminary release of information pertinent to the design and operation of facilities utilizing storable propellants applicable to the Titan II Weapon System.

The information presented herein resulted from a Pre-Qualification Test Program performed on Titan II Propellant Transfer System components under contract to The Ralph M. Parsons Company. The program resulted in the design, fabrication, installation, and operation of a storable propellants facility in order to evaluate propellant ground support components currently available from industry.

The information contained herein basically relates to the following areas of interest:

- Facility Design
- Safety
- Fuel Blending
- Component Handling
- Waste Propellant Liquid and Vapor
- Neutralization
- Component, System and Material
- Compatibility

Although literature surveys and propellant facility tours were conducted prior to design and installation of the Wyle Laboratories facility, problems were encountered during the Propellant Transfer System program relating to operational concepts, personnel safety and materials compatibility. Subsequently, the operational procedures, test methods, etc. were continuously re-evaluated and modified. While the urgency of the program did not permit an extensive investigation of each of the problem areas, information of a significant nature was obtained for inclusion in this Technical Note.

Additional reports will be submitted which will amplify the preliminary information contained herein and which will include additional data obtained during the latter phases of the study program.

The following personnel directed the preparation of this Technical Note:

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ABSTRACT

This Technical Note contains information relative to the design and operation of a storable propellants test facility utilizing nitrogen tetroxide and the Titan II fuel blend as the fluid media. The information was obtained as a result of a Pre-Qualification Test Program conducted for The Ralph M. Parsons Company on Titan II Propellant Transfer System components.

Problems and/or procedures relative to the basic facility utilized, safety aspects, blending of the fuel, general safety concepts, operational procedures, neutralization of waste oxidizer and fuel propellant solutions, and materials compatibility are presented in a preliminary form. Upon conclusion of the study program, additional reports will be prepared amplifying the preliminary information contained herein.

This report has been  
reviewed and approved  
for Wyle Laboratories



L. N. Mortenson  
Program Manager

This report has been  
reviewed and approved  
for the Commander



Langdon F. Ayres  
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SYMBOLS/ABBREVIATIONS USED IN TEXT

|             |   |
|-------------|---|
| Fuel Blend  | Nominal 50-50 blend by weight of unsymmetrical dimethylhydrazine and hydrazine                          |
| $N_2H_4$    | Hydrazine   |
| UDMH        | Unsymmetrical dimethylhydrazine   |
| $N_2O_4$    | Nitrogen tetroxide  |
| $NO_2$      | Nitrogen dioxide  |
| Aerozine-50 | Trade name adopted by Aerojet-General Corporation (nominal 50-50 blend by weight of UDMH and $N_2H_4$ ) |
| Oxidizer    | Refers to storable propellant oxidizer (nitrogen tetroxide)   |
| Fuel        | Used interchangeably with fuel blend  |
| Propellant  | Used interchangeably to describe either the fuel or oxidizer  |
| M. A. C.    | Maximum Allowable Concentration   |

## FACILITY DESIGN

### General

The Wyle Laboratories Storable Propellant Facility is located at Norco, California, approximately 60 miles east of Los Angeles. As shown in Figure 1, the facility is located 1,000 feet from the Cryogenic test area and provides for a 400 foot separation between the area used for fuel blend test operations and the area used for nitrogen tetroxide test operations. The quantity-distance relationship is obtained from Reference 1 of the Bibliography, which specifies that the oxidizer shall be separated from combustible material by a distance not less than 400 feet for quantities of oxidizers over 100,000 pounds.

The nitrogen tetroxide and fuel blend test areas are virtually mirror images with respect to the test slabs, block walls and semi-enclosed test area. The 40 foot x 60 foot open test slabs are separated by 10 foot high reinforced concrete block walls and are sloped to facilitate drainage. The gravity drainage system for each test area diverts propellant and water run-off to a 10,000 gallon recovery tank where the waste fluid is treated with the appropriate alkaline or acidic neutralizing solution. An additional 10,000 gallon water tank is located immediately adjacent to each test area to provide water for flushing and fire control.

Two 6,000 gallon, 347 stainless steel, insulated reservoir tanks are provided for storage of the fuel blend and two similar tanks are provided for nitrogen tetroxide storage. Each tank has a capacity of approximately 6,000 gallons and has a pressure rating of 120 psig. These tanks are also utilized for flow testing conducted with the appropriate propellant.

The fuel blend was obtained by blending UDMH and hydrazine received in 55 gallon drums. The nitrogen tetroxide was provided by tank truck and was transferred directly to the two 6,000 gallon storage vessels.

The open 40 foot x 60 foot test slabs were used for performance of life tests, pressure drop tests, pump calibrations, flowmeter calibrations or other testing requiring a significant flow rate. Three component test cells are located in each functional test building for leakage, cycling or other component operational tests under nominal flow conditions.



FIGURE 1

WYLE LABORATORIES STORABLE PROPELLANT FACILITY

Fuel, Oxidizer and Cryogenic Facility Distance Relationship. Fuel area is at left with oxidizer area in right foreground.

Support facilities, including a main operations building, an oxidizer control blockhouse, main water reservoir tanks, high pressure nitrogen storage bottles, cleaning facilities and general storage areas are located on the 180 foot x 400 foot x 20 foot high hill separating the fuel and oxidizer test areas.

#### Nitrogen Tetroxide Test Area

An aerial photograph of the nitrogen tetroxide test area is shown in Figure 2. The slab area designated by number "6" was used for performance of life tests on pumps where continuous operation under rated flow and pressure conditions was required. The oxidizer was pumped from the 6,000 gallon storage vessel using either a test specimen pump or a Wyle Laboratories pump and, subsequently, through flow measuring sections, flow control valves and applicable pipe sections and finally returned to the tank. Pipe sections in the flow loop were constructed of 304 stainless steel and were mated with nominal 150 pound serrated face flanges. The nitrogen tetroxide was blanketed with gaseous nitrogen to prevent boil-off to the atmosphere and/or contamination of the nitrogen tetroxide by water.

The maximum flow rate obtainable was 300 gpm at line pressures up to 150 psig. The system was instrumented to measure and record the following parameters as required:

- System fluid temperature
- Nominal operational flow rate
- Maximum operational flow rate
- Total discharge head pressure at rated flow
- Shutoff head pressure
- Cavitation characteristics

The center test slab contained a closed loop flow system utilized for performing pressure drop tests on valves, filters, flexible hoses and heat exchangers ranging from 1/2 inch to 4 inches in size. The basic construction and operation of this flow loop is comparable to that of the life cycle flow loop defined above. The system designated by the number "3" in Figure 2 was used in calibration of flowmeters for The Martin Company. This system incorporates a 500 gallon stainless steel tank with suitable inlet and outlet connections for use of the system in conjunction with the

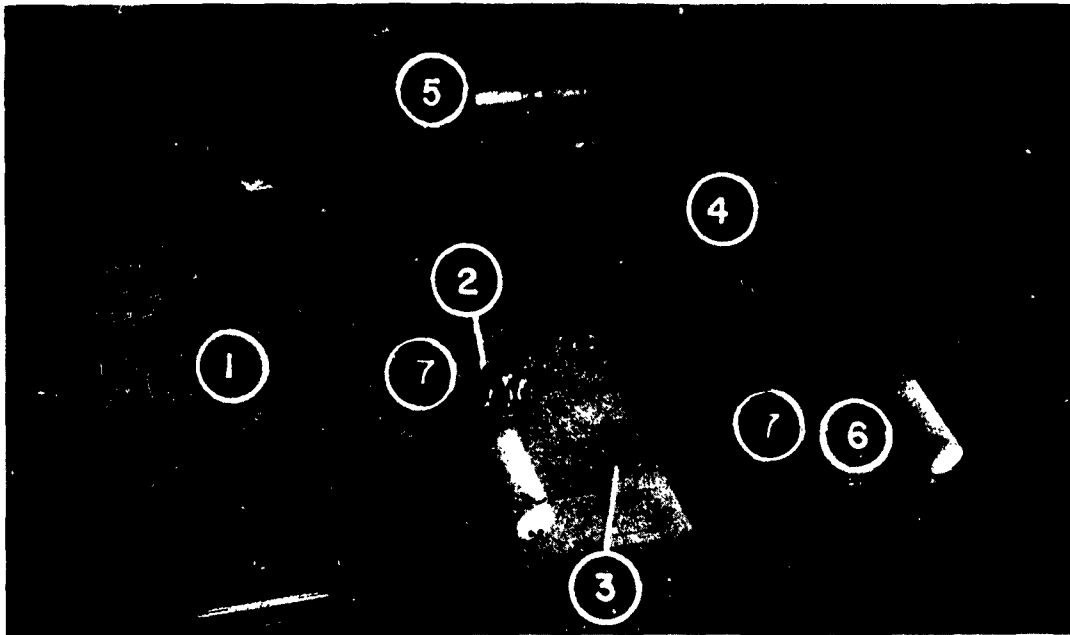


FIGURE 2

TYPICAL STORABLE PROPELLANT TEST AREA  
(Oxidizer Area Shown)

Typical test area layout including test cell building ① ; pressure drop slab ② ; flow calibration system, ③ ; scrubber, ④ ; waste disposal catch tank and disposal basin, ⑤ ; and life test slab, ⑥ . Also shown, ⑦ , are ten-foot high concrete block walls to isolate test slabs.



main 6,000 gallon reservoir tank. A scrubber system designated by the number "4" in Figure 2 was initially installed to neutralize nitrogen tetroxide vent gases and is further described in the body of this Technical Note.

The functional test cells were located under an open-air type roof designated by the number "1" in Figure 2. These test cells incorporated a 75 gallon, stainless steel, 200 psig storage vessel, regulated gaseous nitrogen supply lines, propellant supply lines and test specimen mounting fixtures. The cells were used in performance of internal leakage, proof pressure, cracking and reseal pressure (check valves only), compatibility and life cycle testing of valves, filters, flexible hoses, etc.

An enclosed working area is provided in a portion of the functional test building and was used to facilitate component assembly, disassembly, storage, etc.

Remote instrumentation and control for the nitrogen tetroxide areas was accomplished from the blockhouse located approximately 100 feet from the flow test slabs.

#### Fuel Blend Test Area

With the exception of the scrubber system, the fuel blend test area is essentially a mirror image of the oxidizer test area. Additional high flow water deluge nozzles were incorporated to provide adequate fire fighting and deluge capability. The control station for the life cycle, pressure drop and flowmeter calibration testing is contained in the main building which overlooks the test areas.

## SAFETY

### General

Initial equipment and procedures prior to activation of the storable propellant facility were largely defined as a result of literature surveys and discussions with suppliers. Subsequent experience gained by Wyle Laboratories during performance of the Pre-Qualification Test Program indicates that more specific detailed information should be made available to the missile industry regarding protective clothing, breathing apparatus, atmospheric contamination, etc.

### Protective Clothing

Initially, Gra-Lite limited protective clothing and complete "two-piece" protective suits were obtained for use by personnel during propellant handling operations. The two-piece protective suit (as shown in Figure 3) consisted of a jacket, bib overall, hood, boots and 14" polyvinylchloride gloves. The limited protective clothing consisted of face shields, gloves, boots and aprons. The complete protective suits were used primarily with portable breathing apparatus in handling nitrogen tetroxide. The limited clothing was worn with a canister-type gas mask while handling the fuel blend.

Personnel handling nitrogen tetroxide while wearing the two-piece protective suit developed nitric acid burns around exposed wrists and ankles, due to nitric acid formation resulting from the combination of  $\text{NO}_2$  vapors and skin perspiration. Subsequently, the jacket and overall cuffs were taped to the wrists and ankles, respectively, to prevent acid burns. It is conjectured that a one-piece coverall incorporating a double sleeve design with a tapered inside sleeve and an outer sleeve with zipper would provide better personnel protection. In this case, the gauntlet of the gloves would fit between the two sleeves. The sleeve has the advantage of being removable without opening the zipper and would afford a good liquid seal. However, it has the disadvantage of not providing an absolute vapor seal. The one-piece coverall should incorporate boots sealed to the coverall to effect both a liquid and vapor seal.

The limited clothing was effective in protecting personnel handling the fuel blend. However, in the event of a major fuel blend spill, the complete protective suit should be worn. The complete protective suit afforded



FIGURE 3

COMPLETE PROTECTIVE CLOTHING FOR  $N_2O_4$  TESTING

Technician shown outfitted with two piece Gra-Lite suit, boots, P. V. C. gloves and Scott Air-Pak breathing apparatus. Functional test cells are shown in background.

effective protection to personnel with the exception of the acid burns described above. Personnel wearing the complete protective clothing were uncomfortable, and, due to excessive perspiration, could not wear them much longer than 30 minutes. It is the opinion of Wyle Laboratories that a completely enclosed flexible, light weight all-purpose suit would be extremely desirable for use in handling both the oxidizer and fuel.

Personnel wearing cotton-lined polyvinylchloride gloves experienced dermatitis on their hands when handling the fuel blend. After continuous service, the gloves became discolored, brittle, and cracked, permitting the fuel blend to gradually permeate the gloves. This condition can be prevented by maintenance of the gloves as follows:

1. The gloves should be inspected immediately prior to usage for evidence of excessive wear or cracks.
2. The gloves should be worn no longer than 4 hours.
3. After usage, the gloves should be externally rinsed, turned inside out, again rinsed, and allowed to completely dry before subsequent usage.

#### Portable Breathing Equipment

Initially, Scott Air-Paks, with 15-minute air supply cylinders, were procured for use in handling nitrogen tetroxide. The Air-Paks incorporated a pressure demand regulator to provide a positive air pressure in the face mask which, in turn, prevented toxic vapors from penetrating cracks around the periphery of the mask. Army Chemical Center-approved, Willson canister gas masks, with the designated WIG LG6RTD rocket fuel canister, were obtained for use in handling the fuel blend. Both the canister gas masks and the Scott Air-Paks provided effective personnel protection without exception.

The canister gas masks were used solely with the fuel blend when performing hazardous operations, such as breaking lines, removing components, performance of internal leakage tests, as shown in Figure 4, etc. Scott Air-Paks



FIGURE 4

PROTECTIVE CLOTHING FOR FUEL TESTING

Technician shown conducting test in fuel test cell wearing protective equipment, consisting of apron, P. V. C. gloves, boots and portable Willson breathing apparatus.

were also available in the fuel test area in the event of a major spill. The canister gas masks were not used during handling operations with nitrogen tetroxide, since published literature states that the masks filter out only 80% of the  $\text{NO}_2$  vapors.

Experience with the Scott Air-Paks in handling nitrogen tetroxide indicated that 30-minute air cylinders were more desirable than the 15-minute type to allow sufficient time to deal with emergency situations; i.e., changing ruptured burst discs, replacement of failed valves, etc.

A portable liquid air supply would appear to be highly desirable during prolonged exposure of personnel to heavy concentrations of toxic vapors. The use of a centralized air supply with flexible hoses does not appear desirable due to the limitations of movement of personnel and the possibility of deterioration of the air supply hose.

#### Atmospheric Contamination

Fuel blend vapors were vented to the atmosphere through 20 foot high vent stacks. Nitrogen tetroxide was also vented to the atmosphere both intentionally and as a result of equipment failure. Routine operation of the facility required venting quantities of  $\text{NO}_2$  vapors in excess of those originally anticipated. Filling of the 6,000 gallon storage vessel, component removal, transfer of nitrogen tetroxide, and adjustment of vapor pressure levels in the functional and main storage vessels resulted in the venting of large quantities of vapor to the atmosphere.

Vapor detectors were not used during the program; however, warning horns were used prior to venting to atmosphere and streamers and/or flags were used to indicate wind direction. Operating personnel, when warned by the horn, could note wind direction from the flags or streamers and, subsequently, move upwind of the gases.

The atmospheric conditions; i.e., humidity, wind direction, wind velocity, etc., play an important part in dissipation of the vapors. Under damp and windless conditions, both the white fuel blend vapors and the reddish-brown  $\text{NO}_2$  vapors tend to "hug" the ground. In addition, excessively high humidity and/or fog causes a combination of  $\text{NO}_2$  vapors with atmospheric water vapor, creating a nitric acid mist. In the latter case, personnel exposed to this vapor concentration could be burned.

Higher ambient temperatures and lower relative humidity will result in quicker dissipation of the fuel blend and NO<sub>2</sub> vapors. In this case, the vapors tend to rise, and remain clear of the testing areas.

### Spill Control

Water can be used as an effective agent in controlling fuel blend spills. Removal of fuel spills with water, as shown in Figure 5, proved effective due to the solubility of the fuel in the water. The decrease in vapor formation was immediately evident as a result of the water flushing. The policy of having a water hose on the test slab, during operations which could result in minor spillage, was stressed to all operating personnel.

The use of water to control nitrogen tetroxide spills is considerably different from that required for spills of nitric acid oxidizers such as red fuming nitric acid. In the latter case, water dilutes the existing nitric acid concentration; however, in the case of nitrogen tetroxide, nitric acid is formed by combination with water. For this reason, it is recommended that NO<sub>2</sub> vapors be allowed to stand and boil off in lieu of water flushing. The use of water spray to force fumes away from personnel is sometimes effective in permitting the technicians to continue working on the system during an emergency operation.

The solubility of water in nitrogen tetroxide is only 1.6% by weight. Thus, water flushing does not result in dilution of nitrogen tetroxide, but merely forces the nitrogen tetroxide to a more remote location where it will continue to evolve reddish-brown fumes. Water flushing will also cause the oxidizer to boil off at a substantially higher rate which, in many cases, defeats the purpose of the water usage.

The use of a fine water mist to suppress nitrogen tetroxide boil off has proved effective. The water mist acts in much the same way as fog or extremely humid conditions in reducing the evolution of the NO<sub>2</sub> vapors.

The Waste Propellant Neutralization Section of this Technical Note contains additional details on the effectiveness of water when used to dispose of or control nitrogen tetroxide spillage.

### General Safety Considerations

The following list of recommended safety measures was substantiated during the course of the Propellant Transfer System Program:



FIGURE 5

FUEL SPILL NEUTRALIZATION

Fuel spill on test slab shown being diluted by copious water flushing.



1. Personnel working with storable propellants should wear the following minimum protective clothing: gloves, face shields, aprons, and boots.
2. The cognizant engineer should make sure that the warning system, safety equipment, and waste neutralization system are operative prior to initiating handling operations with the storable propellants.
3. Operating personnel should be aware of local wind conditions and humidity levels prior to performing operations which could result in large quantities of vapors being vented to atmosphere.
4. The use of the "buddy" system is recommended for operations performed under potentially hazardous conditions, or where large quantities of propellants are involved.
5. Operating personnel should be aware of the difficulty in visually observing NO<sub>2</sub> vapors at night, due to the dark, reddish-brown color of the vapor.
6. Some components (e. g., ball valves) should be disassembled under water after exposure to the propellants to avoid sudden release of trapped vapors. The use of cool water will reduce the vapor pressure and afford maximum personnel protection.
7. Water showers and eye wash fountains should be strategically located throughout the facility for use by personnel in case of exposure to the propellants. In the event of a spillage or other exposure, it is important that the propellant be immediately flushed from the affected areas to prevent burns.
8. A qualified physician familiar with the toxicity of the propellants and symptoms resulting from propellant exposure should be retained by the operating agency.
9. Operating personnel should be aware of the physiological symptoms resulting from overexposure to the propellant vapors. Exposure to nitrogen tetroxide may not result in an immediate reaction, but may result in symptoms 10 to 12 hours later.

10. Operating personnel should be aware that continuous inhalation of "tolerable" low-level concentrations of propellant vapors reduces the threshold of sensitivity.
11. All systems used with the storable propellants should be periodically checked for leaks. All components should be pressure checked for external leakage prior to installation in the system.
12. The compatibility of questionable materials should be determined by a "soak" under static conditions prior to installation in the propellant flow loop.
13. Vacuum supports should be provided for burst discs to prevent failures resulting from a negative pressure.
14. All liquid level gauges should incorporate isolation valves which would normally remain closed.
15. Operating personnel should not remove components from a flow loop without following a well-defined procedure for removing all entrapped liquid and vapor within the line.
16. "Check lists" should be prepared prior to initiating propellant handling or test operations.

## FUEL BLENDING OPERATION

### Blending

The storable propellant fuel for use in the Titan II weapon system is an approximate 50-50 blend of UDMH and hydrazine conforming to Aerojet-General Corporation Specification Number AGC-44041B and referred to in the AGC Specification as "Aerozine-50". As of September 1960, no provisions had been made for the delivery of the fuel blend to Air Force contractors. As a result, a large scale UDMH/hydrazine blending system as shown in Figures 6 and 7 was designed for blending approximately 6,000 gallons of fuel blend.

A survey of the available literature indicated that extremely active mechanical mixing of the two fuels would produce a stable propellant mixture which would not stratify during subsequent storage periods. A blending system utilizing pump circulation appeared attractive due to the simplicity of operation and control. The blending system selected was a combination of a pump recirculation technique and a continuous blending operation utilizing the blender shown in Figure 8. The recirculation-blending system consists of a 6,000 gallon tank, two 260 gpm pumps for recirculation and a high turbulence blender as shown in Figure 9.

On September 6 and 7, 1960, sixty (60) drums of hydrazine and sixty (60) drums of UDMH were received at the storable propellant site. During the ensuing two-month period of system design and fabrication, the drums were stored in an open unshaded area. During this approximate two-month storage period, the maximum daytime and minimum nighttime temperatures were 101°F. and 42°F., respectively. No propellant deterioration was noted as a result of the storage period.

Prior to the blending, the 6,000 gallon storage vessel was passivated with a 7% hydrazine and 93% water solution followed by thorough dry nitrogen purging. During the transfer of the UDMH and hydrazine to the 6,000 gallon tank, the drums and associated equipment were electrically grounded. Each drum of UDMH or hydrazine was weighed on a platform scale before and after transfer of the contents, thus providing a measure of the net quantity of propellant transferred into the storage vessel.

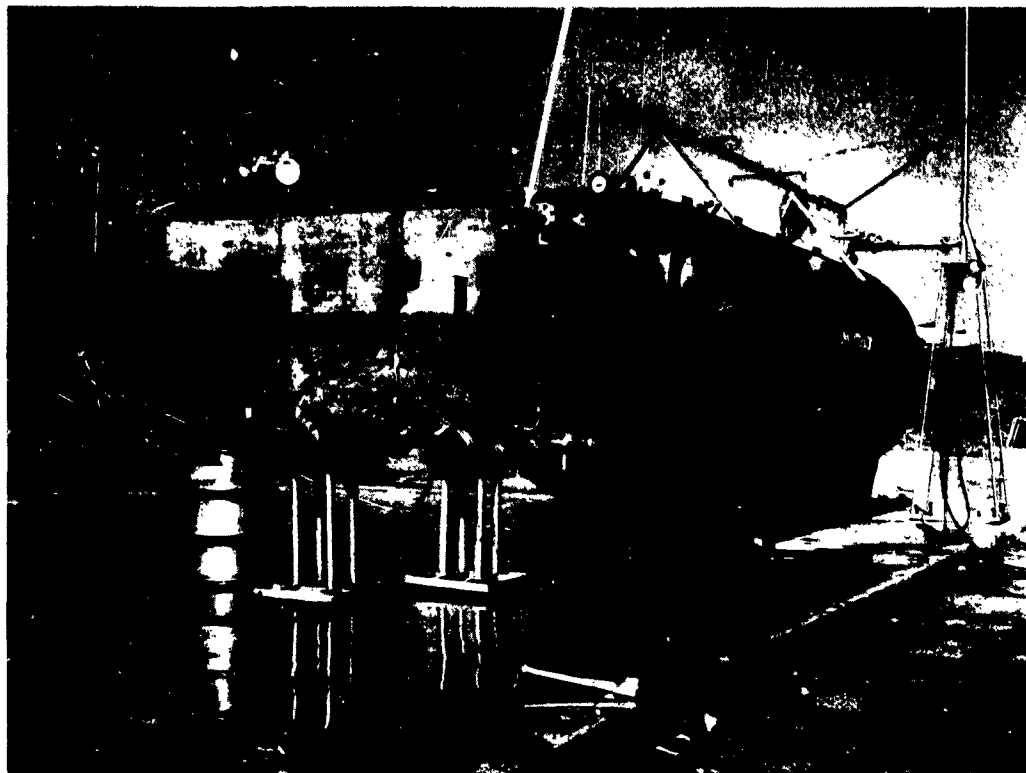


FIGURE 6

BLENDING UDMH/HYDRAZINE

Fuel blending system showing fuel drum, transferpumps, and connecting piping to blender (identified by arrows ).

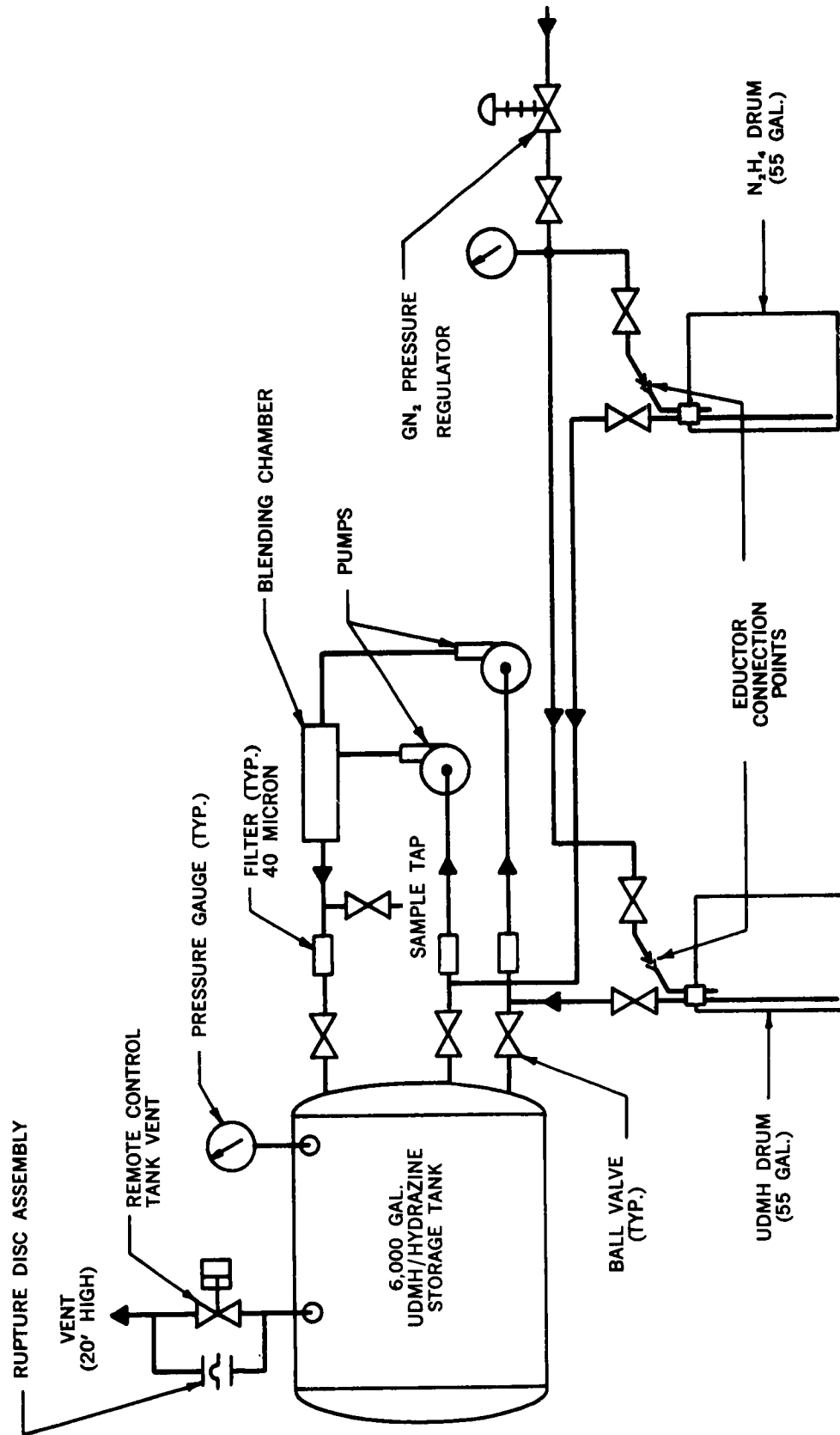


FIGURE 7  
UDMH/HYDRAZINE BLENDING SYSTEM

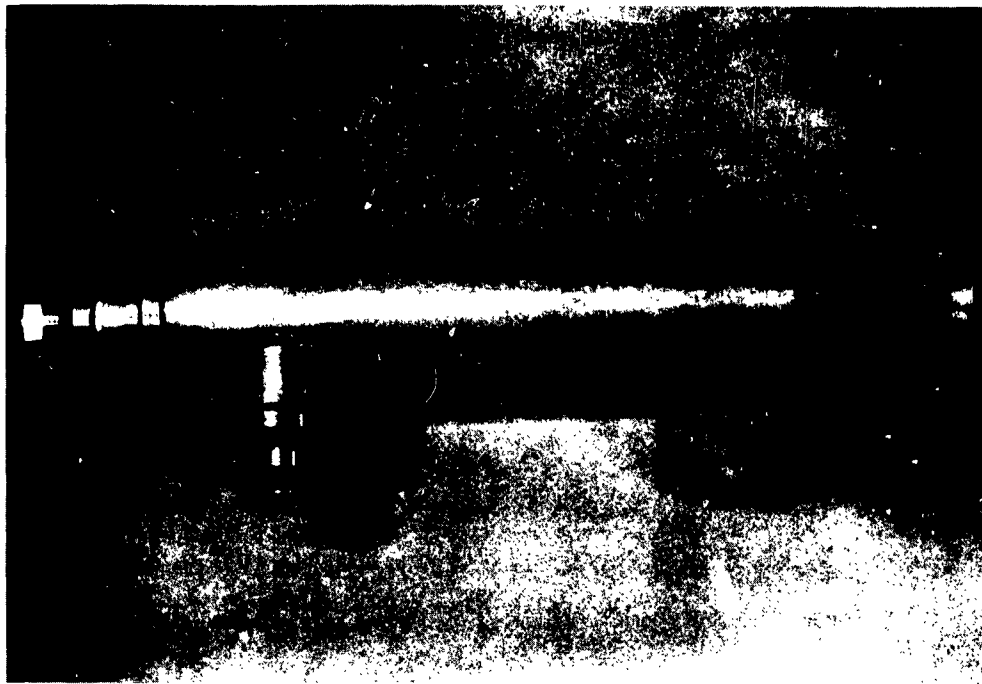


FIGURE 8

UDMH/HYDRAZINE BLENDER

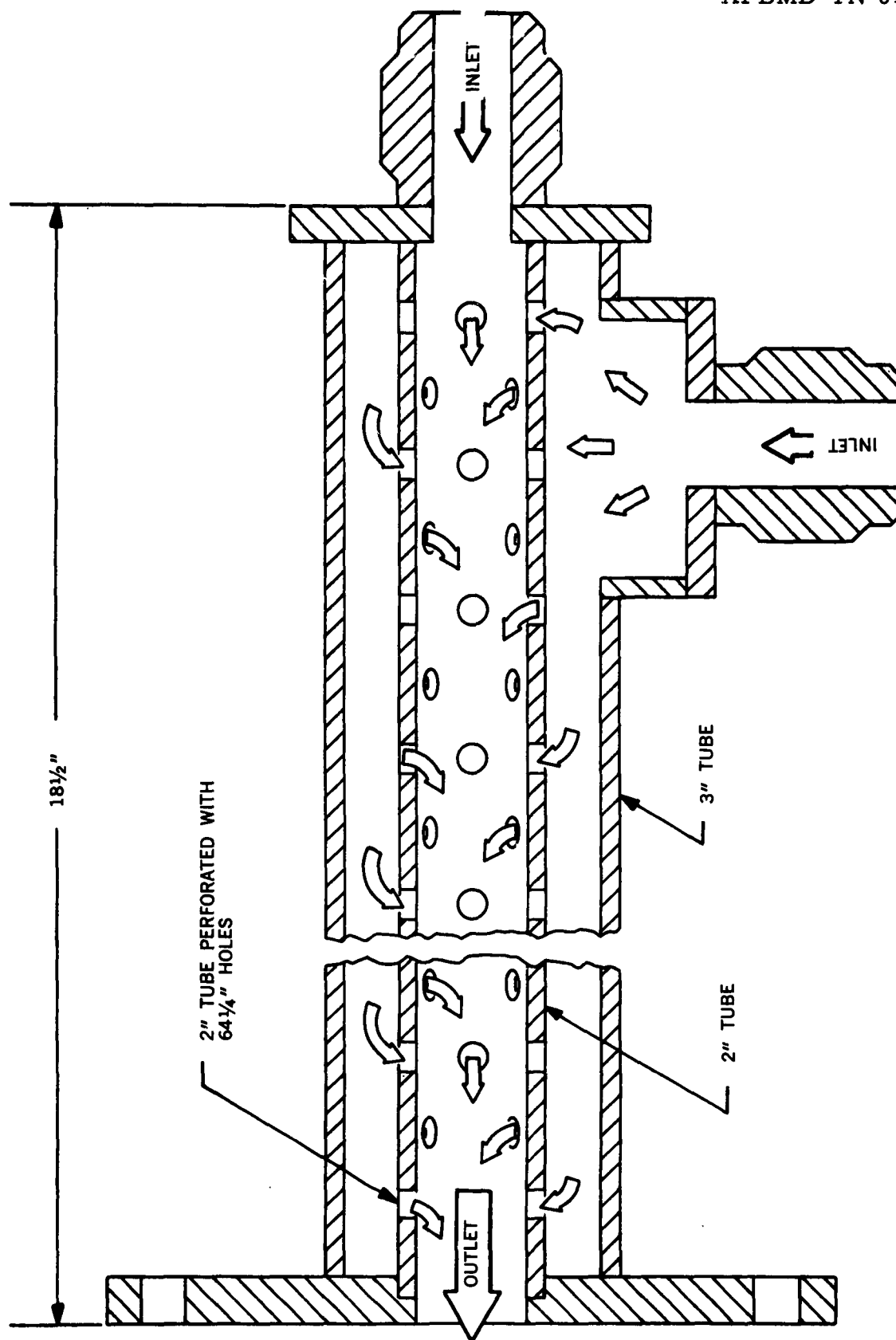


FIGURE 9

UDMH/HYDRAZINE BLENDER DETAIL

The drum unloading was conducted simultaneously from one (1) hydrazine drum and one (1) UDMH drum. Transfer was accomplished using an eductor with a nitrogen transfer pressure of 12 psig applied to the drums (a typical drum was subjected to a proof pressure of 30 psig). Approximately twenty minutes was required to discharge the contents of a pair of drums. This procedure was continued until a total of 20,993 pounds (50 drums) of hydrazine and 20,153 pounds (60 drums) of UDMH were transferred to the storage vessel, resulting in a total propellant weight of 41,146 pounds.

Due to the simultaneous unloading of the UDMH and hydrazine drums, a certain degree of premixing was initially obtained in the 6,000 gallon tank. The UDMH was transferred to the 6,000 gallon tank through the screen covered outlet at the forward end of the tank, as shown in Figure 10. The hydrazine was introduced into a 4 inch diameter pipe which extended nearly the entire length of the tank. The periphery of this pipe contained small diameter holes leading into the interior of the tank.

Following the initial transfer of UDMH and hydrazine into the storage tank, the two 260 gpm centrifugal pumps were used to provide continuous circulation through the blender for approximately one hour. At the end of the first hour of operation, a sample was drawn from the tank for analysis. The pumps were again started and operated for an additional one hour and fifteen minute period, during which time samples were drawn from the tank at fifteen minute intervals. The temperature and specific gravity measurements recorded during this sampling period are shown below:

| <u>Specific Gravity<br/>of Mixture</u> | <u>Temperature<br/>°C</u> | <u>Specification<br/>Requirements</u> |
|--|---------------------------|---------------------------------------|
| 0.907                                  | 15                        | 0.9073 $\pm$ 0.0042                   |
| 0.905                                  | 15                        | 0.9073 $\pm$ 0.0042                   |
| 0.905                                  | 16                        | 0.9063 $\pm$ 0.0042                   |
| 0.904                                  | 17                        | 0.9054 $\pm$ 0.0042                   |
| 0.903                                  | 18                        | 0.9046 $\pm$ 0.0042                   |

During the recirculation period, the propellant was withdrawn through the 6 inch diameter screen covered and the 4 inch diameter perforated pipe tank outlets and subsequently pumped through the blender and returned to



the tank through an upper 4 inch diameter line which also traversed the entire length of the tank. The returned mixture was subsequently distributed throughout the entire length of the tank into the vapor phase of the tank. Because of the unique tank withdrawal and return line configurations, considerable internal turbulence should have occurred in the tank; therefore, the success of the blending operation can probably be attributed to a combination of the internal tank configuration and the blender.

#### Temperature Rise of Fuel Blend with Water

An exothermic reaction of the fuel blend with water was noted. Thus, precautions were required to insure that no moisture remained after rinsing the thermometer, hydrometer and beaker used to obtain the specific gravity and temperature measurements. The temperature rise resulting from the exothermic reaction of the fuel blend with water was investigated. The results, as shown in Figure 11, indicated that a maximum temperature rise of 77 F. is obtained with the fuel blend and water mixtures at 28% and 37% water by weight contingent upon the test procedure being utilized.

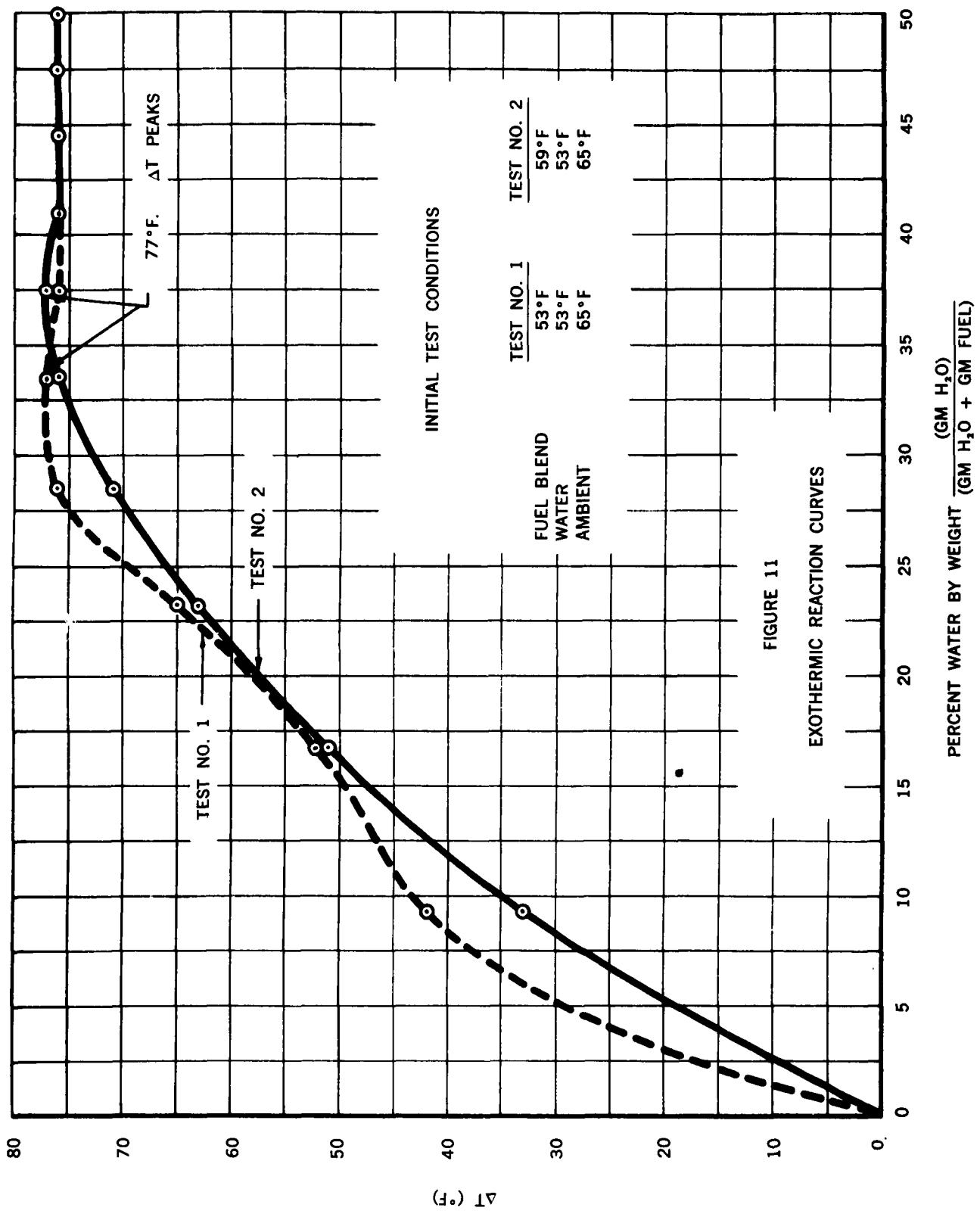
For Test No. 1, the fuel blend was poured into an insulated glass Dewar flask. After recording the stabilization temperature, water(10% by weight with reference to the original quantity of fuel blend) was added incrementally and the corresponding temperature rise associated with each incremental addition of water noted. For Test No. 2, each incremental addition of water was made to similar quantities of undiluted fuel blend, e.g., after each incremental addition of water to the fuel blend the peak temperature was recorded and the solution discarded. The test was then resumed using additional Dewar flasks containing the same initial quantity of undiluted fuel blend.



FIGURE 10

FORWARD END OF 6,000 GALLON FUEL TANK

Forward interior of tank showing screen covered 6 inch diameter outlet for UDMH and 4 inch diameter outlet for hydrazine.



## COMPONENT HANDLING

### Removal of Components from Propellant Systems

Large numbers of components were installed and subsequently removed from the propellant test systems during the Pre-Qualification Test Program. A notable contrast exists between those procedures utilized for removal of components from cryogenic or conventional fuel systems, and procedures developed for use with storable propellants. In the latter case, the removal operation is complicated due to the toxicity of storable propellants, and the ability of many components to retain entrapped propellants after removal from the system.

During the initial facility and system design, provisions were made to include aspirators, nitrogen purge connections, liquid drain valves and isolation valves for each test section used for the test components. Special protective clothing, safety equipment, and component contamination procedures were all required in facilitating expedient removal and handling of the components.

Procedures used to facilitate removal of the components from the fuel and oxidizer systems were similar with the exception of the type of protective clothing and breathing apparatus and the use of aspirators for evacuation of the oxidizer test section.

### Fuel Blend Component Removal

The reservoir tank pressure was first reduced by venting to a low value. The isolation valve on the storage tank outlet was closed and the fuel in the test section was forced into the 6,000 gallon tank through the tank return line by applying gaseous nitrogen pressure to the test section purge connection. In some instances, where a gas phase existed in flow lines adjacent to the test section, both the reservoir tank outlet and return block valves were closed and the fuel forced from the test section into the adjacent piping by again applying gaseous nitrogen pressure to the purge connection. The isolation valves on either side of the test component were then closed and the applied gaseous nitrogen pressure was relieved.

The above procedure was not sufficient to remove all of the fuel and quantities of fuel blend (up to 2 quarts) often spilled during the component removal process. Therefore, personnel engaged in the component removal wore protective gloves, boots, aprons, face shields and canister gas masks. After removal of the component, the test section mounting flanges were covered with mating blind flanges or polyethylene sheeting.

The component was immediately flushed with copious quantities of water until such time that it could be handled by operating personnel without protective gloves or breathing apparatus. Further component decontamination procedures are contained in the Component Decontamination Section of this Technical Note.

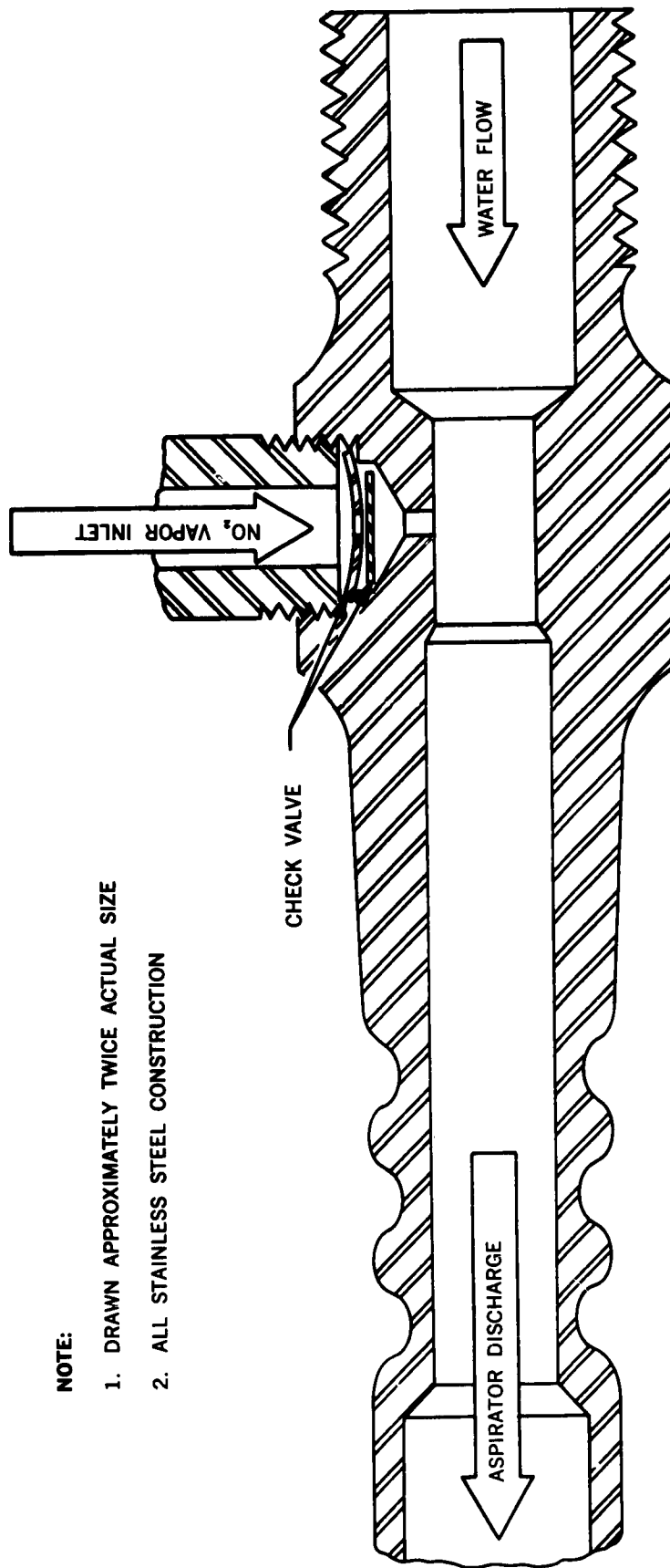
#### Nitrogen Tetroxide Component Removal

The gaseous nitrogen purge procedure outlined above was also used in removing nitrogen tetroxide test components. However, water aspirators, as shown in Figure 12, were used to further evacuate the test section prior to removal of the component.

The isolation valves on either side of the component were closed and the test section evacuated by using a 1/4 inch line attached to the venturi of the aspirator. The aspirator subsequently removed residual nitrogen tetroxide from the test section and discharged the waste propellant into a 12 inch drain manifold illustrated in Figure 13. The aspirator was capable of evacuating the test section or a complete flow loop to approximately 3 inches of Mercury absolute. It was determined that the aspiration time could be decreased by utilizing a nitrogen purge to "boost" the entrapped liquid and vapor through the aspirators.

The test section was considered to be free of entrapped nitrogen tetroxide when the  $\text{NO}_2$  vapors being discharged from the aspirators decreased to a nominal value. The component was then removed from the test section and flushed with water.

Personnel involved in the removal of nitrogen tetroxide components wore complete protective equipment, including full Gra-Lite suits, boots, polyvinylchloride gloves, and Scott Air-Paks. Component removal in a major flow loop or in the functional test cells was performed in a similar manner.



**NOTE:**

1. DRAWN APPROXIMATELY TWICE ACTUAL SIZE
2. ALL STAINLESS STEEL CONSTRUCTION

FIGURE 12  
TYPICAL NITROGEN TETROXIDE ASPIRATOR

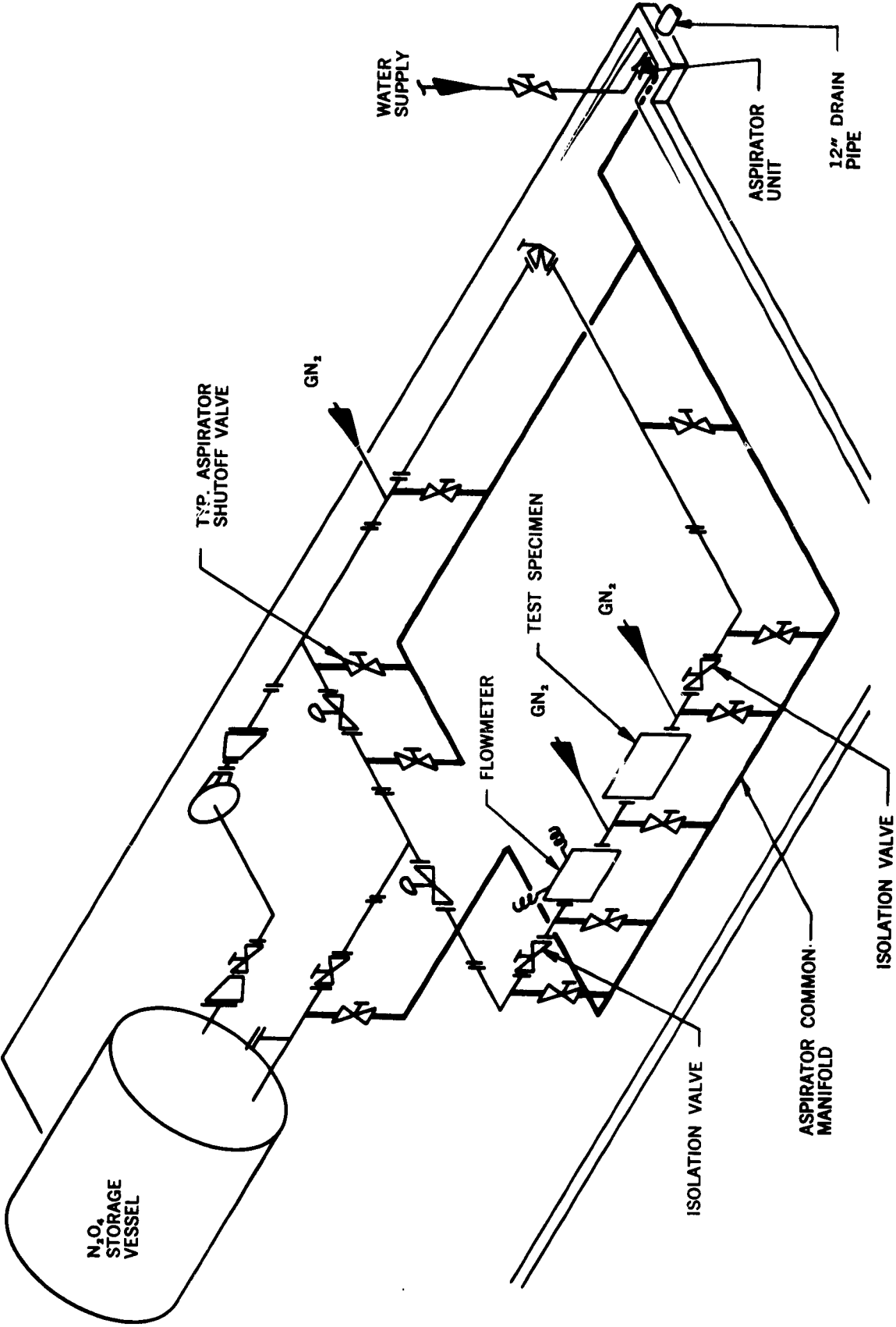


FIGURE 13  
ASPIRATION SYSTEM

Water aspirators were not used in removing fuel blend components due to the following reasons:

1. The time required to evacuate the test section was considerably longer due to the low vapor pressure of the fuel blend.
2. Fuel spillage was readily soluble in water and was easily flushed away.
3. The quantity of vapors emanating from spilled fuel was considerably less than that quantity resulting from an identical quantity of oxidizer spillage.

Compon      Decontamination

As a result of the Pre-Qualification Test Program, it was determined that a definite problem exists in decontaminating components which have been exposed to either nitrogen tetroxide or fuel blend. For the purpose of this discussion, a decontaminated component is considered that component which meets the following criteria:

1. Propellant odors cannot be detected.
2. Black light and visual examination reveal no indication of propellant contamination.

Initially, all test components were disassembled and cleaned in accordance with a modified "LOX cleaning" procedure. After exposure of the component to propellant service, the need for effective decontamination varied, contingent upon the component's performance history. Components which complied with all operational requirements would normally be decontaminated prior to shipment to the vendor. In this case, the primary reason for decontamination was to eliminate the possibility of personnel exposure to toxic vapors possibly retained within the components. Those components which failed to meet the operational requirements, and which were subsequently modified by the vendor, also required decontamination to prevent personnel exposure to toxic vapors. In the latter case, however, the problem was complicated due to a Pre-Qualification Test Program requirement that the components be recleaned in accordance with the LOX cleaning



procedures prior to retest; thus, the component was required to pass the visual and black light inspection criteria contained in the cleaning procedure.

Components removed from the propellant test systems were thoroughly flushed with tap water to facilitate handling of the component without wearing protective gloves or breathing apparatus. If this procedure was not followed, the quantity of liquid or vapors entrapped in the component would necessitate excessive handling precautions. The component was then taken to the cleaning area and subjected to a general decontamination process as follows:

1. The component was initially immersed in containers filled with de-ionized water. Separate containers were used for components exposed to the fuel blend and nitrogen tetroxide.
2. The component was then rinsed with trichlorethylene.
3. The component was dried with gaseous nitrogen.

Propellant liquid and/or vapors would often remain entrapped in manually-operated ball valves and, to an extent, in pumps and flowmeters, even though the above procedures were followed.

The ball valves were periodically operated through full travel limits while submerged in the de-ionized water. This procedure was required due to the inherent design features of this type of valve. As shown in Figure 14, typical ball valve construction permits propellants and vapors to escape from the valve in the 1/4-closed position only. It is of interest that the entrapped liquid and vapors were often released with sufficient violence to constitute a personnel hazard, although the operation was performed with the ball valve submerged.

Some of the components were partially or completely disassembled under water, due to the difficulty in removing entrapped vapors. This was particularly true of components which had a porous cast body material, complex flow passages, or teflon seats or seals. When possible, teflon seats and seals were removed to prevent personnel exposure to toxic vapors emanating from the teflon. A detailed discussion of this problem is contained in the Material Compatibility Section of this Technical Note.

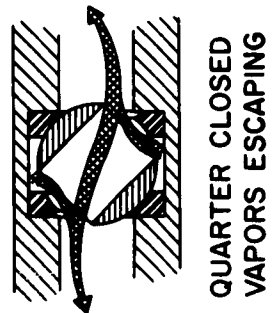
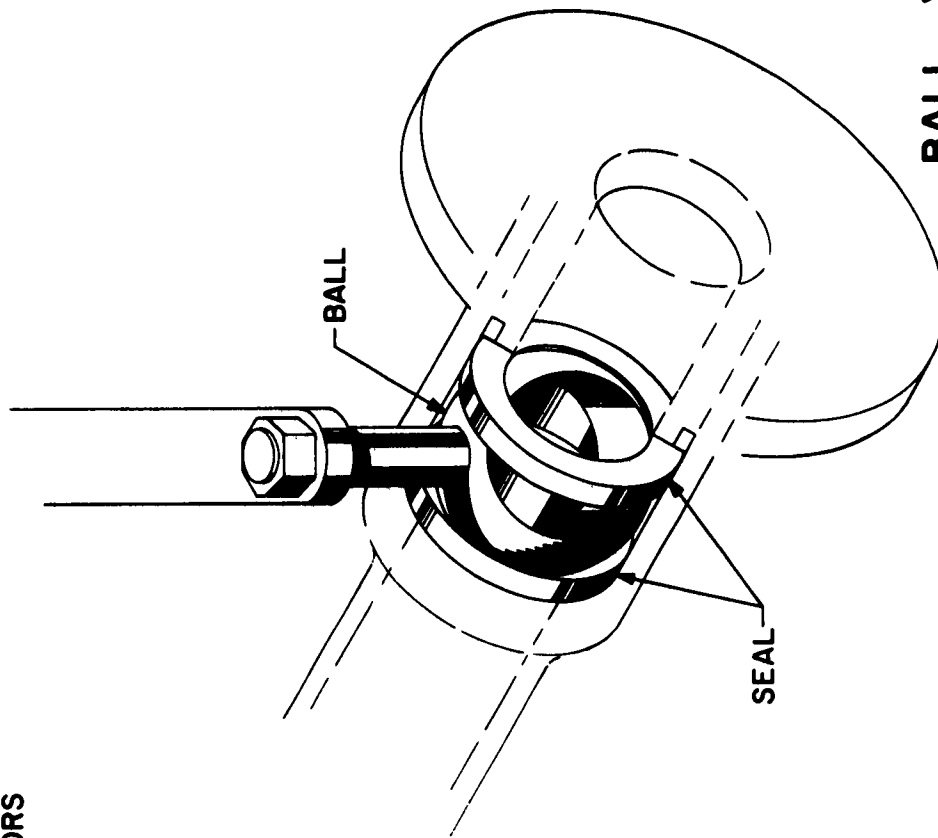
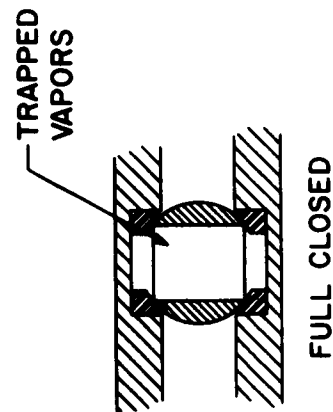
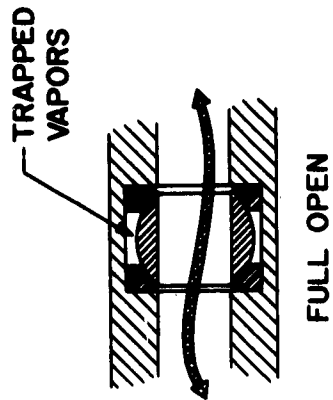


FIGURE 14  
BALL VALVE CONSTRUCTION

Initially, it had been assumed that components were satisfactorily decontaminated by following procedures outlined in the preceding paragraphs. Experience early in the program indicated that this was not the case, particularly for those components exposed to the fuel blend. The fuel blend apparently permeates pump bearings and cast body materials which have a relatively high degree of porosity. Contamination of the atmosphere above the M.A.C. level caused by unpackaged fuel blend components, supposedly decontaminated, was repeatedly observed. This atmospheric contamination was particularly noticeable when components were stored in closed areas for any length of time after testing. Contamination levels were substantially higher if the unpackaged component contained teflon.

Although numerous attempts were made to decontaminate assembled components, a satisfactory method could not be established. Additional investigation of decontamination procedures for assembled components would appear to be desirable.

## WASTE PROPELLANT LIQUID AND VAPOR NEUTRALIZATION

### Fuel Blend

Fuel vapors were vented directly to atmosphere through a 20 foot high 2 inch vent stack mounted on the main 6,000 gallon storage vessels. Typical venting operations of the 6,000 gallon tank resulted in white vapor plumes approximately 15 feet in length.

Each of the concrete slab test areas were sloped to facilitate flushing fuel spills into a concrete drain basin located at the end of each slab. The basins were manifolded to a 12 inch pipe which was, in turn, connected to the top of a 10,000 gallon mild steel collection tank. This tank was located at a grade level lower than the propellant storage and test slab area and was vented directly to atmosphere.

The run-off from the slab areas primarily consisted of water used to flush down the slabs for cleaning purposes. Minor fuel blend spills resulting from test specimen removal were flushed into the drainage system with water. The collection tank contents, when checked for pH value, indicated minor fuel blend contamination resulting in a weak basic solution.

In the event a litmus paper test indicated a basic pH value, the collection tank contents were neutralized by the addition of a nominal quantity of citric acid granules. These granules were flushed into the tank through the concrete drain basins, using water. The action of the flushing water flowing into the collection tank generally provided sufficient mixing. Additional granules were added until the contents of the tank had been neutralized, as indicated by subsequent pH tests.

### Nitrogen Tetroxide

The drainage system for the nitrogen tetroxide test area was identical to that provided for the fuel blend area. A vapor scrubber was installed for scrubbing NO<sub>2</sub> vapors vented from the storage tank or other portions of the test systems. This scrubber, as illustrated in Figure 15, was a 2-stage mild steel unit which incorporated provisions for recirculating 30 gallons of 20% sodium hydroxide (NaOH) solution through each stage. As



FIGURE 15

NO<sub>2</sub> VAPOR SCRUBBER

Original two stage configuration illustrating 500 gallon H<sub>2</sub>O/NaOH storage tank (horizontal), venting stack, and vapor inlet line (lower right hand corner).

illustrated in Figure 16, the vapors entered the first stage, subsequently passed through the Roschig tubes and on through the second stage, where the process was repeated before the associated gases were vented to atmosphere. As  $\text{NO}_2$  vapors passed through each stage, the sodium hydroxide solution was sprayed down upon the Roschig tubes in an attempt to neutralize the rising vapors. The Roschig tubes served to increase the contact surface to volume area of the  $\text{NaOH}$  solution and  $\text{NO}_2$  vapors.

After facility activation, the first stage and the connecting plumbing was attacked by an acid solution as a result of inadvertent venting of  $\text{NO}_2$  vapors directly through the mild steel unit with the recirculating sodium hydroxide system inoperative. As shown in Figure 17, several holes developed in the first stage unit and the scrubber system became inoperative.

The interior of the remaining stage was plastic coated with "Permabar" in an effort to prevent additional acid attack on the mild steel. After a few weeks of use, the second stage also developed minor leaks, possibly due to incorrect application of the coating. While the basic problem resulted from not using a compatible material (i.e., stainless steel) in the scrubber construction, there is still some doubt as to the scrubber's effectiveness.

With a highly basic solution discharging through the shower heads, the  $\text{NO}_2$  vapors were still not satisfactorily neutralized during venting operations. This condition possibly resulted from insufficient Roschig tube contact area or possibly from insufficient "contact time" for the  $\text{NO}_2$  to form  $\text{HNO}_3$  and subsequently react with the basic solution.

With the scrubber system inoperative, several alternative methods were tried in an attempt to neutralize the oxidizer vent gases. These alternative methods are described in the following paragraphs.

The 2 inch common vent line was positioned horizontally and a 10 foot section of 1/2 inch diameter stainless steel tubing was inserted into the vent line. The 1/2 inch diameter tubing incorporated 5 sets of 1/4 inch holes drilled around the periphery of the tubing and spaced approximately 2 feet apart. During venting operations, water was discharged through the 1/2 inch diameter tubing in an attempt to cause the  $\text{NO}_2$  vapors to go into solution. Residual  $\text{NO}_2$  vapor concentration levels resulting from these attempts were unacceptable.

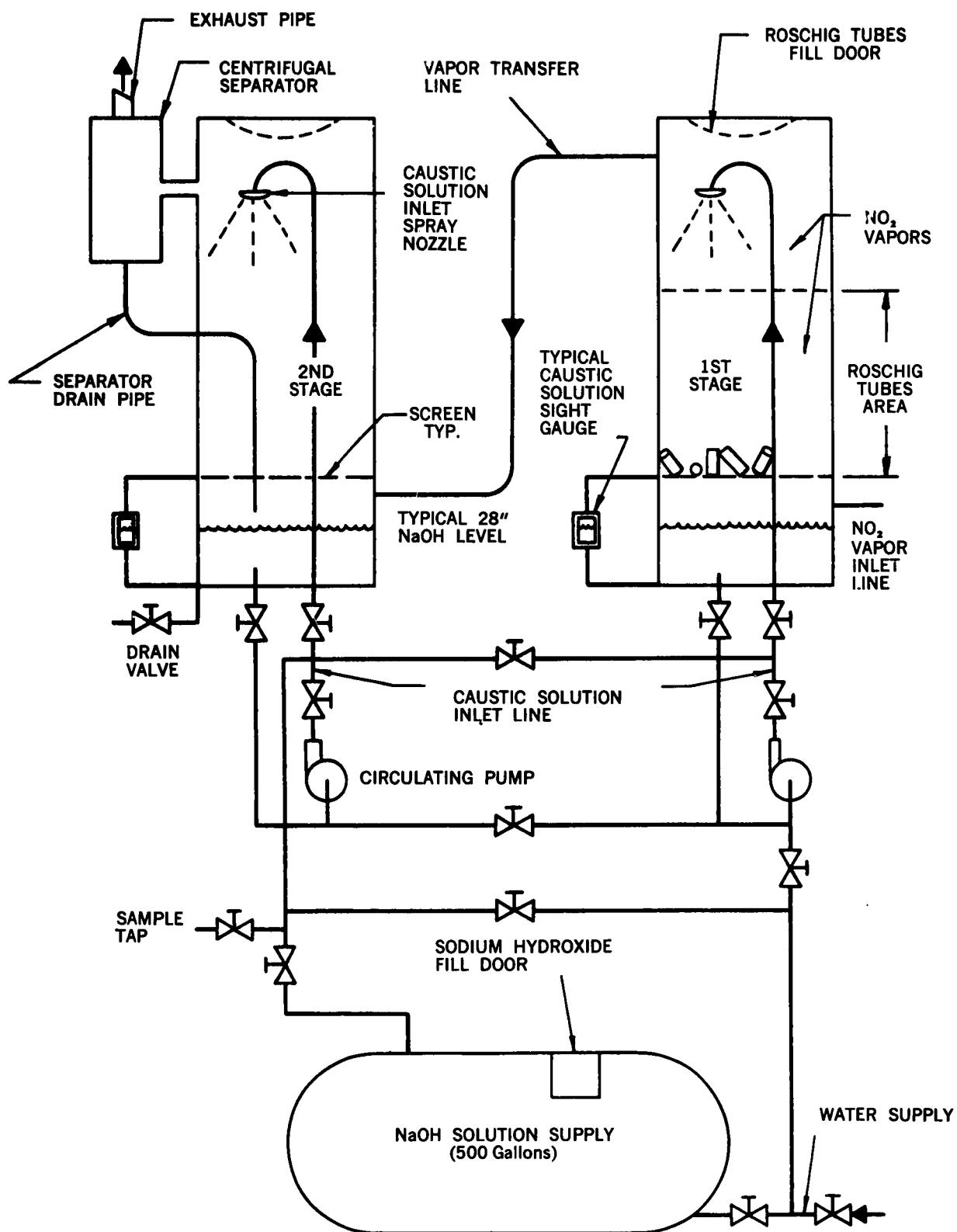


FIGURE 16

SCHEMATIC OF ORIGINAL  $N_2O_4$  SCRUBBER



FIGURE 17

INOPERATIVE VAPOR SCRUBBER

First stage of mild steel scrubber illustrating the results of acid attack due to  $\text{NO}_2$  vapor venting with the alkaline recirculating system inoperative.



Similarly, an attempt was made to "scrub" the NO<sub>2</sub> vapors with water only, utilizing a single stage of the scrubber system. In this case, the water was not recirculated, but was allowed to drain out of the scrubber into the 10,000 gallon stainless steel collection tank. Again, the solubility of the NO<sub>2</sub> vapors in the water spray was insufficient, resulting in unsatisfactory performance.

Vapor neutralization was also tried without recirculation using one stage of the scrubber system and a 20% caustic soda solution. After exposure of the solution to the NO<sub>2</sub> vapors, the solution was again allowed to drain into the 10,000 gallon collection tank. This method was used for approximately 3 weeks during which time the venting cycle durations were varied over a wide range of storage vessel pressures. Complete vapor neutralization was achieved only when the storage tank pressure was below 10 to 15 psig. At higher storage vessel pressures, the excessive volume of NO<sub>2</sub> vapors resulted in unsatisfactory performance of the scrubber.

The alternative methods described above were not preceded by a thorough analysis, but were primarily used in an attempt to provide some relief from the NO<sub>2</sub> vapors in an expedient manner. The initial scrubber system utilized was inadequate, particularly from the standpoint of fast response time to high flow rate, short duration venting. The same problem would probably exist using any conventional scrubber system unless an extremely high capacity unit was used.

The dilute acidic solutions in the 10,000 gallon collection tank were initially neutralized by the addition of sodium hydroxide. Due to the limited capacity of the collection tank, several catch basins were excavated and sodium hydroxide added to the basin waste solution in order to maintain a neutral pH value.

During the Pre-Qualification Test Program, minor oxidizer spills frequently occurred, particularly during component removal. Since these spills seldom exceeded a gallon, they were immediately flushed with water to the drainage system into the catch basin. A major nitrogen tetroxide spill did not occur during the course of the program.

As noted previously, the addition of copious amounts of water to nitrogen tetroxide spills results in an increased evolution of toxic vapors. NO<sub>2</sub> vapors evolving from nitrogen tetroxide spills in the semi-enclosed test cell areas were often controlled with a fine mist-like spray. The use of the spray served only to minimize the evolution of the vapors, however.

## COMPONENT, SYSTEM AND MATERIAL COMPATIBILITY

### Nitrogen System Contamination

A 37,500 cubic foot high pressure bottle bank was used as a gaseous nitrogen source. As shown in Figures 18 and 19, all of the nitrogen cylinders were manifolded to provide a high volume supply for both the fuel and oxidizer test areas. The possibility of cross-contamination was considered remote, due to the expected high system working pressure and continuous use of the supply system.

As a result of pressurization tests on an empty 6,000 gallon nitrogen tetroxide storage vessel, the nitrogen supply cascade became depleted. A 4-hour period elapsed before the cascade was refilled. When the pressure tests of the oxidizer storage vessels were resumed, fuel odors emanated from the nitrogen pressurization line. It became apparent that fuel vapors had permeated the nitrogen manifold supply lines to the oxidizer test area. This cross-contamination undoubtedly resulted from the low nitrogen supply pressure.

To preclude the possibility of cross-contamination, the gaseous nitrogen supply cylinders were manifolded into two banks of nine each. One system supplied nitrogen to the fuel test area, while the other supplied nitrogen to the oxidizer test area. Thus, the nitrogen supply systems for each area were completely isolated, as shown in Figure 18.

After the modifications were effected, the nitrogen supply system and the "contaminated" oxidizer storage vessel were evacuated to 3 inches of Mercury absolute for a 24-hour period to preclude the possibility of entrapped fuel vapors remaining in any portion of the oxidizer supply system.

During the initial facility design and installation, isolation valves were installed in most cases throughout the nitrogen system in the oxidizer test area to prevent NO<sub>2</sub> vapors from backing up into the nitrogen supply lines. In several cases, where the check valves were not installed, NO<sub>2</sub> vapors backed up into the nitrogen supply lines, resulting in failure of hand regulators and/or pressurization valves.

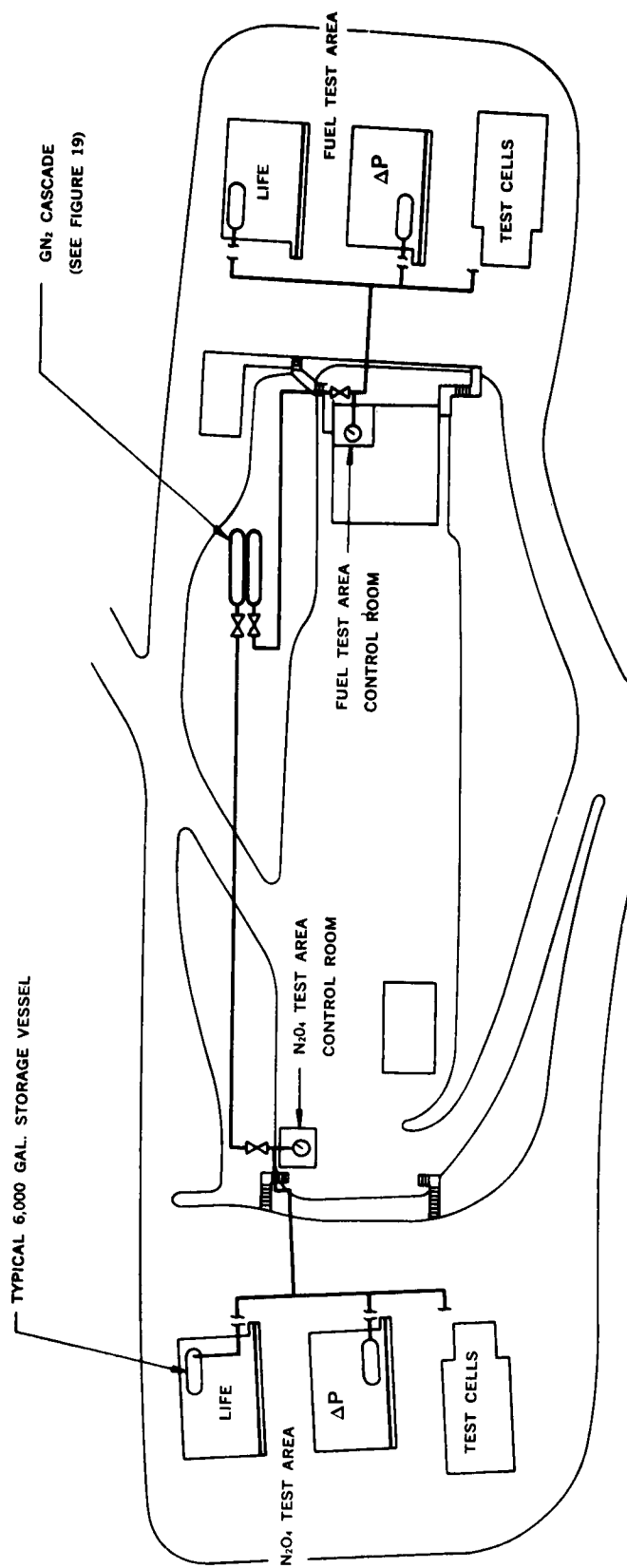


FIGURE 18

## GASEOUS NITROGEN DISTRIBUTION SYSTEM

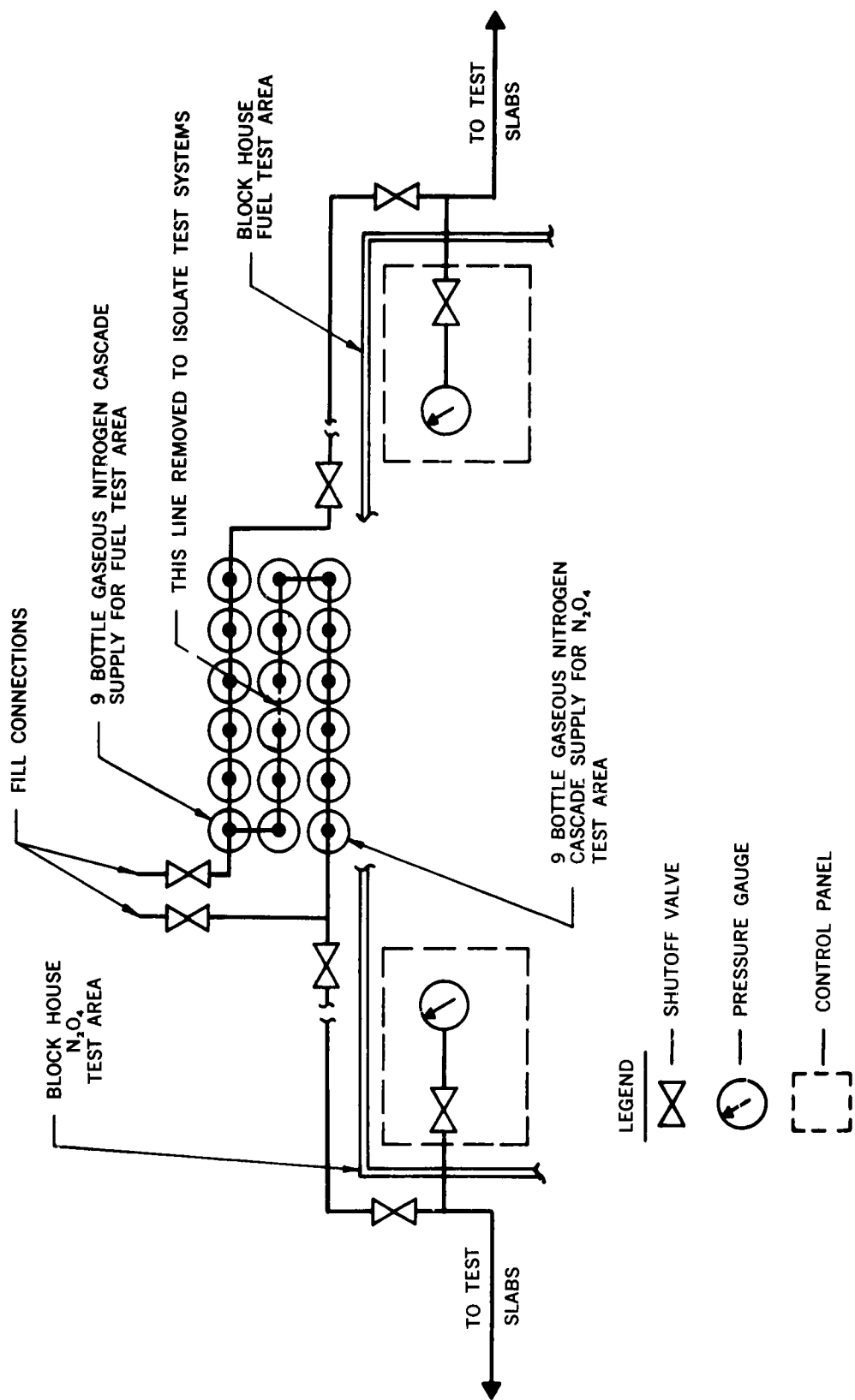


FIGURE 19

GASEOUS NITROGEN STORAGE CASCADE

In the first instance, NO<sub>2</sub> vapors attacked the nylon seats and brass bodies of four, 3-way Asco solenoid pressurization valves and the valves became inoperable. Subsequently, check valves were incorporated into the pressurization system downstream of the solenoid valves.

In the second instance, the NO<sub>2</sub> vapors attacked the nylon and brass portions of the Victor Equipment Company hand regulators. The results of the NO<sub>2</sub> corrosion are shown in Figure 20. Again, check valves, installed in the pressurization system downstream of the regulators, prevented recurrence of the failures.

#### Nitrogen Tetroxide System Leakage

Severe leakage problems were frequently encountered due to the difficulty in achieving effective seals at mating flanges and/or seals used in the oxidizer systems.

Following component installation in a flow test loop, and after all flanges were tightened, the system was pressure checked with nitrogen. No detectable leakage was observed. Subsequently, when the same flow loop was used for nitrogen tetroxide service, the seals at the mating flanges would leak. Retorquing of the flange bolts normally stopped the nitrogen tetroxide leakage. However, either minor leakage or heavy outgassing from the teflon gaskets resulted in a noticeable NO<sub>2</sub> odor in the proximity of the flow loops. It was conjectured that the cold flow of the teflon gaskets was responsible for the above leakage. Additional work is warranted in this area, due to the toxicity of the NO<sub>2</sub> vapors prevailing in the vicinity of the flow loops.

AN fittings which exhibited no tendency to leak when pressurized to 250 psig with gaseous nitrogen subsequently leaked in nitrogen tetroxide service when subjected to pressures of only 30 psig. This apparently is a common problem, and several missile contractors recommended that preliminary pressure checks be accomplished with helium in lieu of nitrogen.

Further, it was recommended that the flared surface of the tubing and the beveled edges of the fittings be polished to eliminate nicks or scratches which could result in potential nitrogen tetroxide leakage.

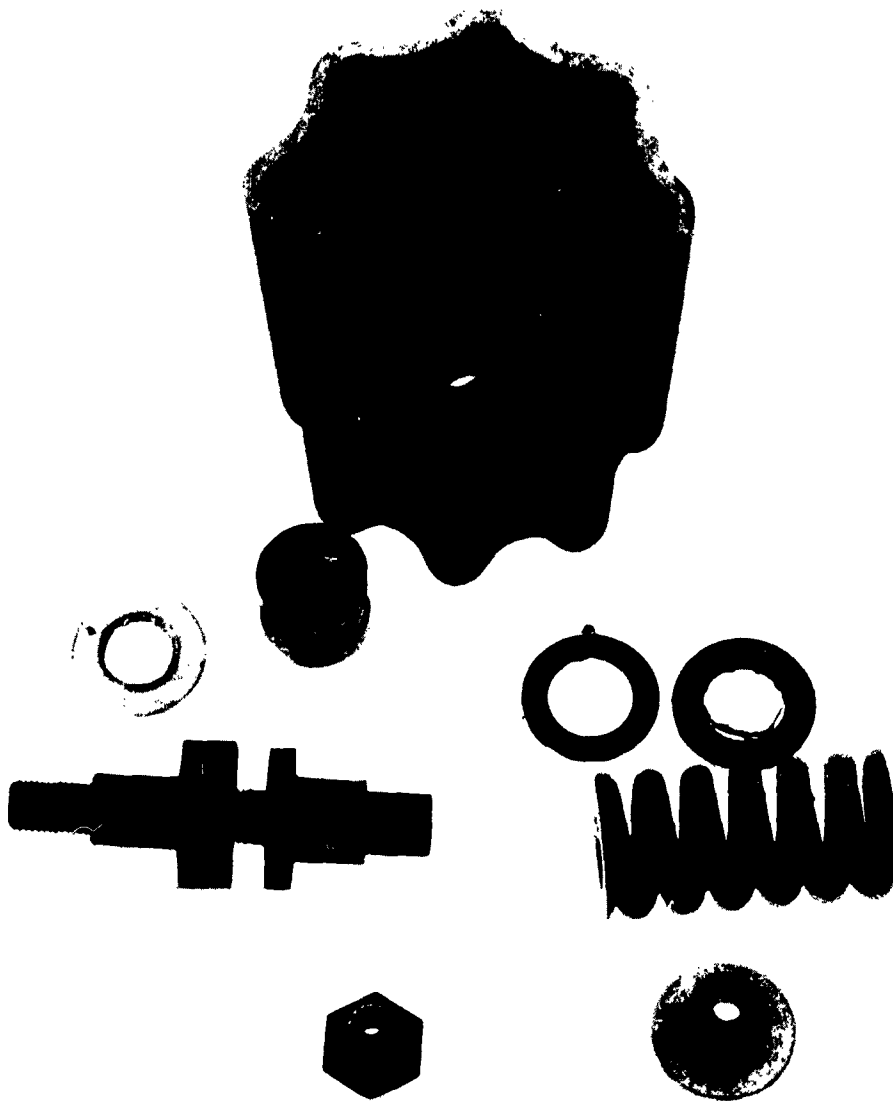


FIGURE 20

CORRODED GN<sub>2</sub> REGULATOR

Nitrogen hand regulator which failed due to corrosive attack of NO<sub>2</sub> vapors on steel spring, bearing race, brass and nylon seats, seals, etc.

As of this date, the effectiveness of polishing the tubing and fittings, and in conducting pressure checks with helium has not been substantiated; however, these methods would appear to offer means of overcoming the leakage problem.

Nitrogen tetroxide has, on occasion, been observed to permeate some metal castings under pressure. It appears that the propellant literally "leaks" through the casting metal. It is recommended that quality control procedures be effected to minimize casting porosity.

During operation of the facility at ambient temperatures above 70°F., NO<sub>2</sub> leakage is considerably more prevalent than that encountered on cooler days. This phenomenon is undoubtedly due to the low boiling point of nitrogen tetroxide.

#### Lubricants

No satisfactory lubricant combining good lubricating qualities with propellant compatibility has as yet been found. As a result, galling often occurred with threaded internal stainless steel parts, as well as with external nuts and bolts. Literature surveys indicated several lubricants to be compatible with both storable propellants. Due to the expedient nature of the Pre-Qualification Test Program, a decision was made to use the recommended lubricants in lieu of conducting extensive tests which would potentially result in an "ideal lubricant. The results obtained from using the recommended lubricants are briefly summarized as follows.

DC-11, which appears on many compatibility lists, performed unsatisfactorily throughout the Pre-Qualification Test Program. The DC-11 lubricant deteriorated after less than 12 hours of continuous exposure to the oxidizer, leaving a gritty-particle residue. While DC-11 is apparently incompatible with nitrogen tetroxide, no deterioration was noted when the lubricant was used with the fuel blend. However, the lubricant, if used excessively, has been observed to absorb the fuel blend and subsequently outgas with attendant personnel hazards. DC-11 was not effective to any great extent in preventing galling of stainless steel parts.

KEL-F grease, also recommended in current literature, has a tendency to dissolve rapidly in nitrogen tetroxide, leaving a "mushy" residue. KEL-F lubricant was not used with the fuel blend.

Because of the limited lubricant life characteristics of DC-11 and KEL-F grease, both lubricants required replacement after a limited number of cycles in propellant service.

Flaked graphite was tried, but difficulty was encountered in maintaining a lubricant bond between the rotating parts of test components. Due to the necessity for continuous reapplications, usage of this lubricant was discontinued.

Additional evaluation tests are currently being performed on other lubricants, and the results will be included in subsequent reports.

#### Sealants

Water glass (silicate of soda) and flake graphite are listed in literature as a compatible sealant for nitrogen tetroxide service. Several instances were noted where a 50-50 mixture of sealant appeared to be incompatible. However, one application indicated satisfactory compatibility during extended use.

The first failure of the water glass-flake graphite occurred when leakage was noted around a sealed teflon gasket on a manhole cover for one of the 6,000 gallon reservoir tanks. In this case, however, the reservoir tank had been subjected to pressure reversals ranging from 3 inches of Mercury absolute to nearly 100 psig.

Water glass-flake graphite was also used on a second 6,000 gallon storage tank in a similar application to that described above. In this application, no leakage was observed over a 4-month period.

The second failure condition occurred when a leakage indicator failed as shown in Figure 21. The indicator incorporated a



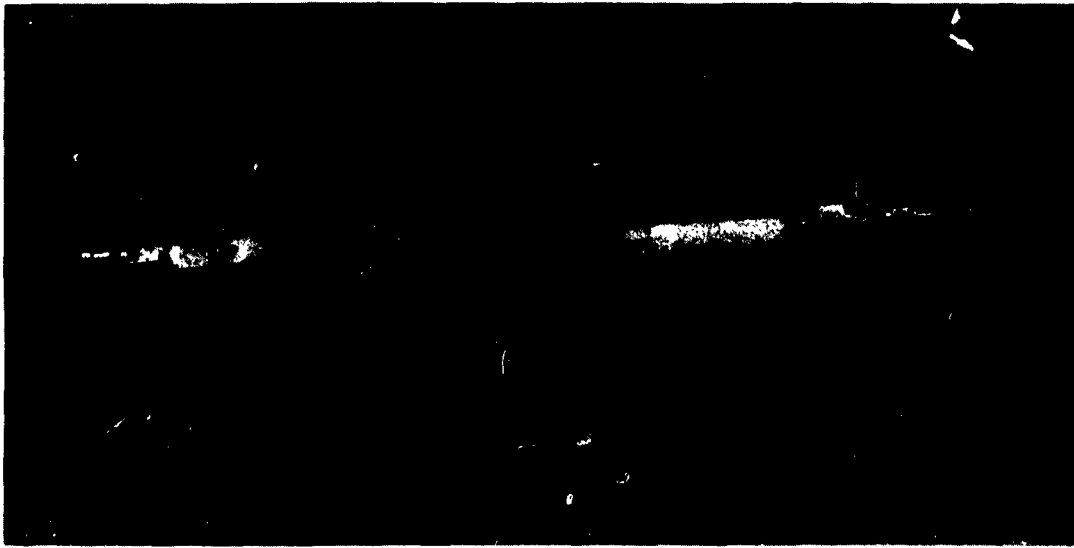


FIGURE 21

RUPTURED  $N_2O_4$  LEAKAGE INDICATOR

Failure resulting from the possible inter-action between the combined use of polyethylene tubing and water glass-flake graphite sealant.

glass burette sealed to polyethylene tubing with water glass-flake graphite. Figure 22 illustrates a fragment of the ruptured unit containing a powdery form of the water glass-flake graphite sealant, indicating possible material incompatibility. In addition, prior to the failure condition, water glass-flake graphite was observed downstream of the leakage indicator in a nitrogen tetroxide test system.

The incompatibility of polyethylene tubing is discussed elsewhere in this Technical Note. However, the indicator failure may have been caused by inter-action between the polyethylene tubing and the water glass-flake graphite sealant.

It is concluded that water glass-flake graphite should be used cautiously as a sealant for nitrogen tetroxide service until such time as definite proof of its compatibility can be established.

#### Teflon

Although teflon is chemically inert, certain grades of teflon are extremely porous to nitrogen tetroxide and, to a lesser extent, to the fuel blend. The fact that many grades and types of teflon are available was learned during the course of the program when problems arose concerning leakage, component decontamination and toxicity. Gaskets fabricated from 25% glass-filled teflon were the only type that proved to be satisfactory from the standpoint of cold flow, leakage and outgassing.

Types of Teflon - A sintered powder form of teflon is available in at least three grades -- Teflon 1, 5, and 7. Teflon 7 is the highest grade teflon in that there is less void content and, therefore, less permeability than the lower grades of Teflon. Teflon 5 and 7 were not tested during the program so, consequently, there is no data available. Teflon 100 differs from the sintered powdered Teflons in that it is an extruded, compression molded thermoplastic and, according to literature references, less subject to permeation than the sintered powdered Teflons.

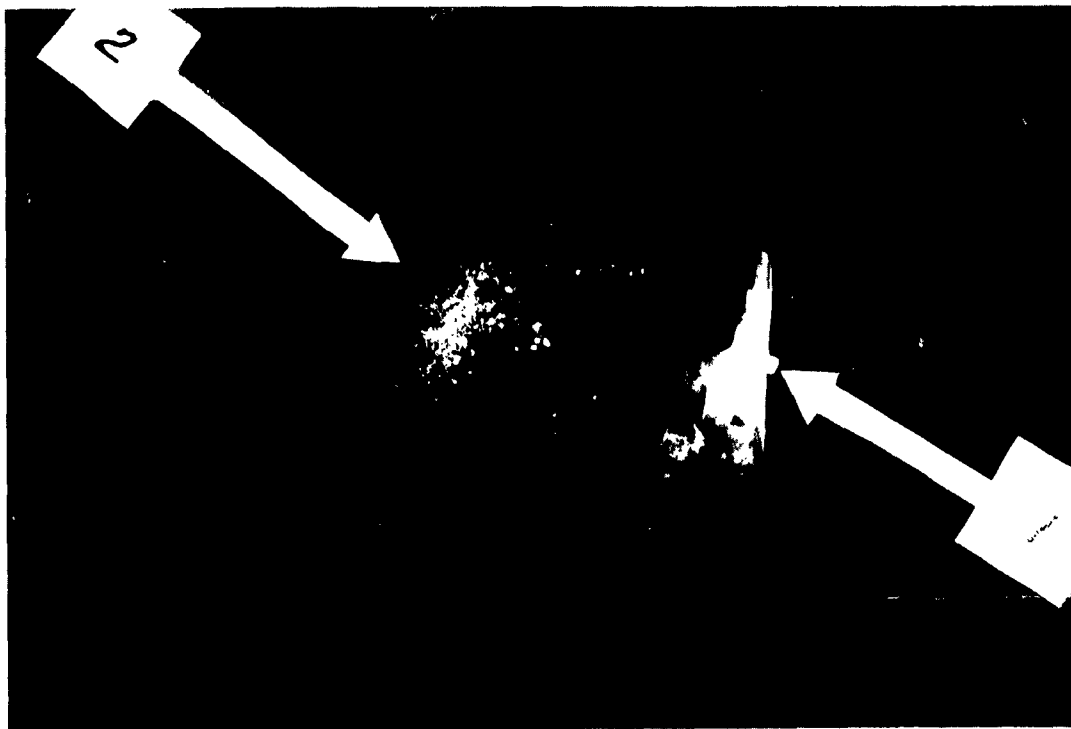


FIGURE 22

$N_2O_4$  LEAKAGE INDICATOR FRAGMENT

Fragment of ruptured indicator showing crystallized and powder form of sealant mater (items 1 and 2 respectively) following service with  $N_2O_4$ .

Permeability of Teflon - During the test program, Teflon 1 gaskets, seals and seats that had been exposed to both nitrogen tetroxide and fuel blend retained sufficient quantities of the propellant to burn the skin during disassembly and inspection of the component.

Teflon gaskets exposed to nitrogen tetroxide were discolored in a circular area extending approximately 1 inch from the inside diameter of the gasket. The material was subsequently subjected to a decontamination procedure consisting of a de-ionized water soak for 8 hours, a trichlorethylene rinse and, finally, a gaseous nitrogen purge. The Teflon gasket was then sealed in a polyethylene bag. In addition to the discoloration, NO<sub>2</sub> fumes (reddish brown in color) outgassed from the gasket and inflated the polyethylene bag as shown in Figure 23. Teflon 100 gaskets were exposed to the propellants during the program but did not show any permeation improvement over the Teflon 1 type.

Teflon 1 gaskets which had been exposed to the fuel blend and decontaminated in the same manner as described for the nitrogen tetroxide gaskets, were also observed to outgas after being sealed in plastic bags. There was no discoloration of the gaskets, but typical ammonia vapors were observed, and the polyethylene bags became inflated.

The outgassing problems outlined above could be particularly serious from the standpoint of inexperienced personnel opening the sealed bags and releasing the toxic fumes.

Decontamination Problems with Teflon 1 and Teflon 100 - Additional efforts to decontaminate the gaskets subjected to nitrogen tetroxide exposure were made by heating them in an oven at approximately 200°F for 4 hours at ambient pressure, and at 160°F for one hour at near vacuum conditions (vacuum degassing). These efforts were unsuccessful since the heating process caused the gaskets to become completely permeated with the reddish-brown color (somewhat lighter than the original discoloration ring).

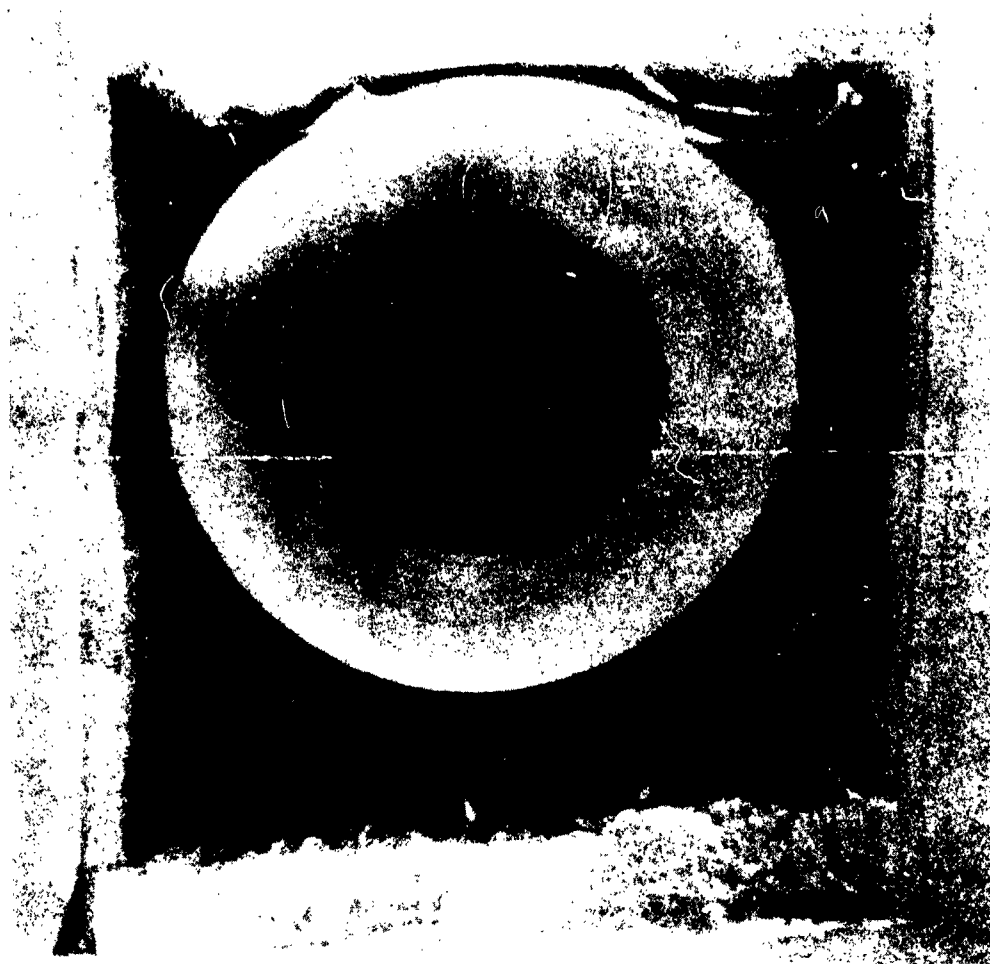


FIGURE 23

PERMEATED TEFLON GASKET

NO<sub>2</sub> outgassing within sealed bag following unsuccessful attempt to decontaminate permeated teflon gasket used in oxidizer flow loop.

Successful decontamination of Teflon 1 and Teflon 100 gaskets was finally achieved by exposing the gasket to direct sunlight for approximately two days. It is conjectured that the sunlight was responsible for the disappearance of the propellant discoloration initially observed on the gaskets. A more expedient decontamination process under controlled conditions is highly desirable, and the decontamination process warrants further investigation.

Results with Other Types of Teflon - Attempts were made to install stainless steel and Teflon spiroallic gaskets in both the fuel and oxidizer flow loops. During installation, the serrations on the flanges did not intermesh with the serrations on the spiroallic gaskets, causing system leakage under pressure. Therefore, this type of gasket is not recommended for use with serrated flanges.

Teflon impregnated asbestos gaskets, when installed in a nitrogen tetroxide test system, leaked by capillary action. Gaskets exposed to the propellant and subsequently subjected to the cleaning procedures previously described, were distorted and discolored and also outgassed. Therefore, Teflon impregnated asbestos gaskets are not recommended for storable propellant usage.

25% Glass-Filled Teflon - Experimental gaskets fabricated from 25% glass-filled Teflon were installed in both fuel and oxidizer flow systems. Contamination and outgassing of the gasket material was extremely nominal after exposure to the propellant. The gaskets exhibited no trace of either propellant after being subjected to the cleaning procedure previously described.

The 25% glass-filled Teflon has proved to be the most satisfactory gasket material and is recommended for use in gaskets, O-rings, seals, etc. The use of this type of Teflon should substantially reduce personnel hazards in handling components which have been exposed to storable propellants.

Teflon Tape - Teflon tape performed satisfactorily as a sealant when used on pipe threads. It is compatible for both fuel and oxidizer usage, but has the disadvantage of shredding on disassembly.

### Manhole Cover Seals

Problems were encountered in effecting sealing of the manhole covers used on the 6,000 gallon fuel blend reservoir tanks, as shown in Figures 24 and 25. Initially, teflon seals were used with manhole covers; however, leakage occurred approximately 12 hours after the storage vessel was filled with the fuel blend. The teflon seals were subsequently removed and replaced as follows:

One vessel was sealed with a Raybestos Manhattan, Inc. No. 670 sealing material which was cut to size from 1/8 inch sheet stock. This material does not presently appear on any of the fuel blend compatibility lists.

The second vessel was sealed with a Stillman Rubber Company No. 613-75 seal (Butyl rubber), also cut to size from 1/4 inch sheet stock. This material appears on present compatibility lists.

The Teflon seals removed from the 6,000 gallon tanks exhibited no indication of material decomposition or transverse scratches. It is conjectured that the leakage resulted from the cold flow characteristics of Teflon and/or poor mating surfaces on the vessel and cover. No evidence of leakage or seal deterioration has been observed during the 4-month period that the replacement seals have been installed.

The problems encountered in sealing the manhole covers on the oxidizer storage vessels are discussed in that section of this Technical Note entitled "Nitrogen Tetroxide System Leakage".

### Sight Gauge and Leakage Indicator Failures

Polyflow tubing, which is a trade name for polyethylene tubing, was used for liquid level gauges on the 75 and 6,000 gallon nitrogen tetroxide storage vessels due to the tubes' translucent characteristics. The tubing was flared for use with AN fittings to facilitate installation.

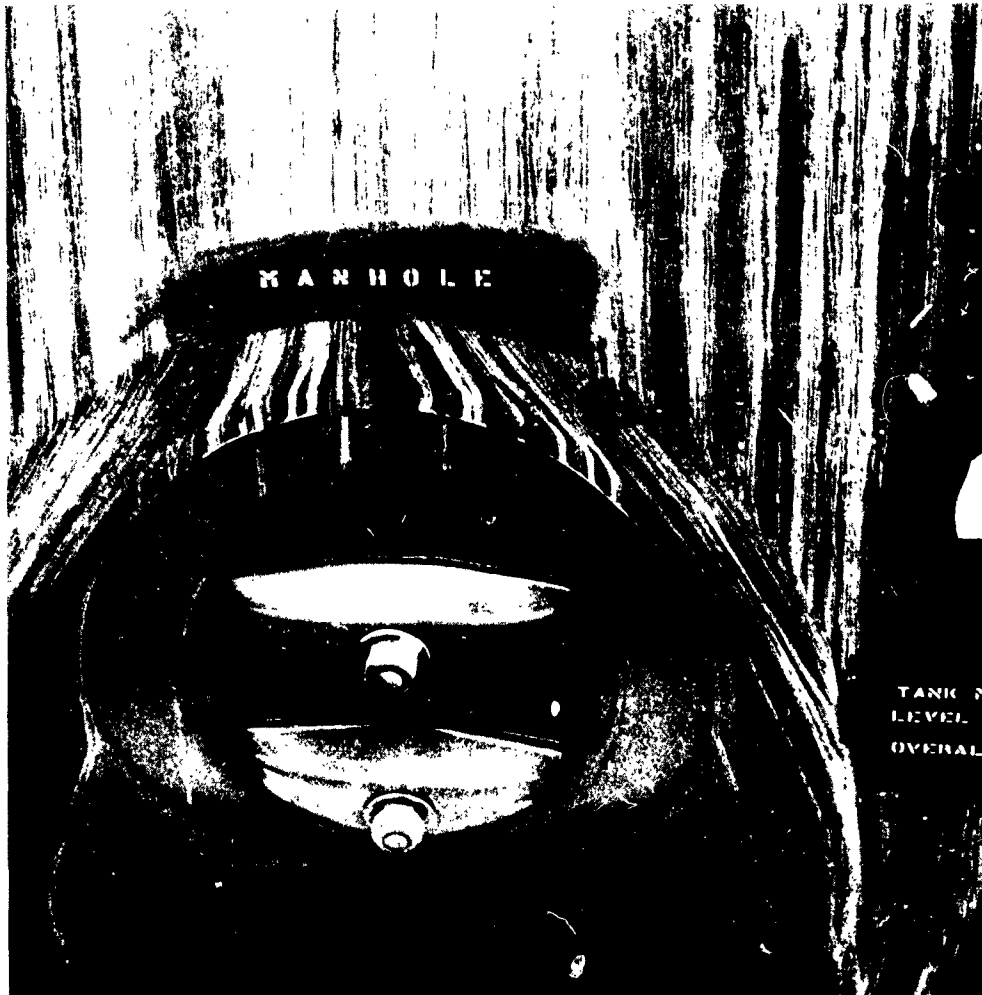


FIGURE 24

MANHOLE COVER--PROPELLANT STORAGE VESSEL

Typical manhole cover installation on 6,000 gallon propellant vessel.



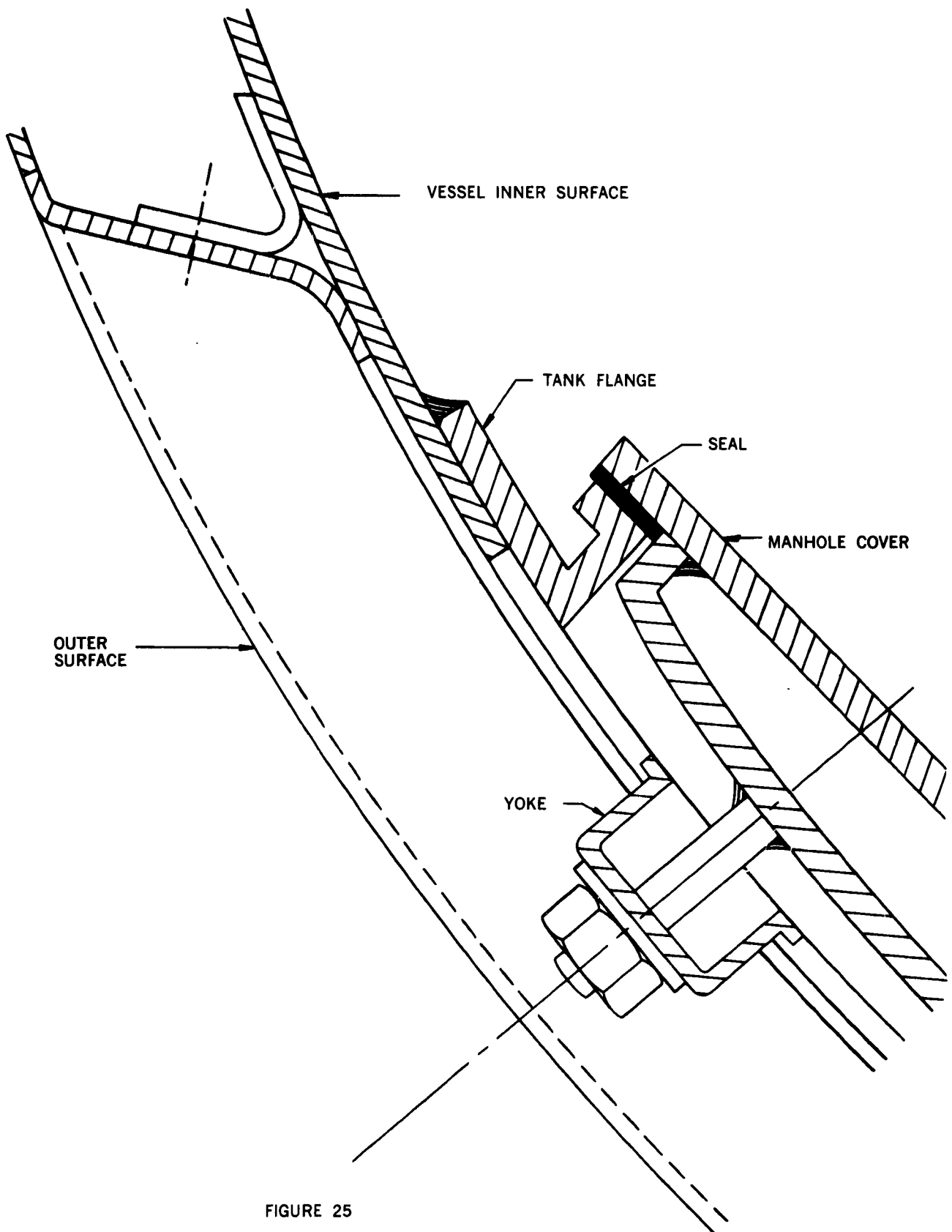


FIGURE 25

CROSS SECTION MANHOLE COVER

Failure of one of the indicator tubes used on a 75 gallon storage vessel occurred within a few days after exposure to nitrogen tetroxide. The failure occurred during a nitrogen tetroxide transfer operation under a pressure of approximately 20 psig. This pressure was well within the tube's rated pressure. Visual examination of the tubing after failure revealed that the tubing was permeated with nitrogen tetroxide and, in addition, contained many fine cracks, as shown in Figure 26.

As a result of this failure condition, all sight gauge tubing was periodically replaced every 3 days until a functionally compatible replacement was located. As an interim measure, Teflon 1 tubing is currently being used for liquid level gauges. While this tubing has the disadvantage of NO<sub>2</sub> outgassing, no line ruptures have been experienced to date.

Leakage Indicators - Leakage indicators were originally fabricated from a 1/2 inch diameter graduated glass burette connected to stainless steel tubing by means of polyethylene tubing. One end of the polyethylene tubing was flared for use with an AN fitting, while the other end was inserted over the burette and sealed with water glass - flake graphite and a clamp.

During the functional test, one of the leakage indicators ruptured. Examination of the failed unit shown in Figure 21, indicated that the burette had pulled out of the polyethylene tubing. Further observation of a burette fragment, shown in Figure 22, indicated that the sealant had hardened and crystallized and could be easily removed by a slight scraping or rubbing action. Examples of the hardened and crystallized sealant are identified as Items 1 and 2, respectively, in the aforementioned figure.

To preclude the possibility of failure recurrence, leakage indicators containing protective pyrex outer sealant and swage lock sealing features were installed. These indicators, as shown in Figures 27 and 28, have performed satisfactorily to date.



FIGURE 26

POLYETHYLENE TUBING

Small specimen of polyethylene tubing taken from liquid level indicator.  
Note fine cracking and discoloration due to  $N_2O_4$  permeation.

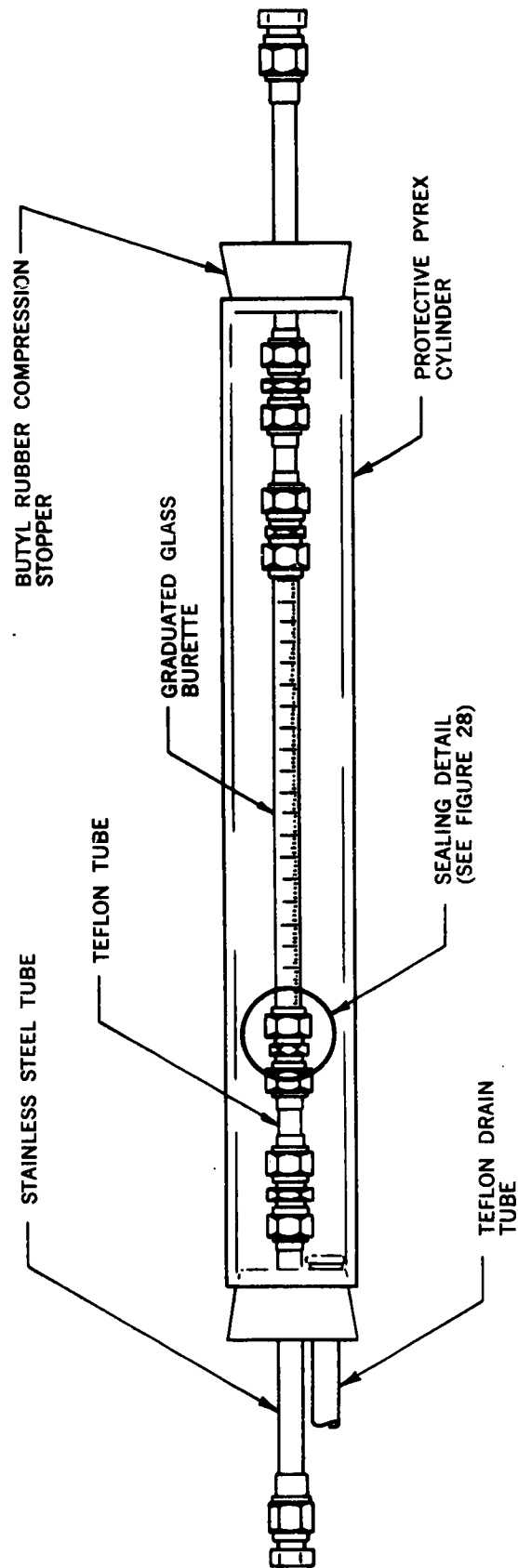


FIGURE 27  
N<sub>2</sub>O<sub>4</sub> SPECIMEN LEAKAGE INDICATOR

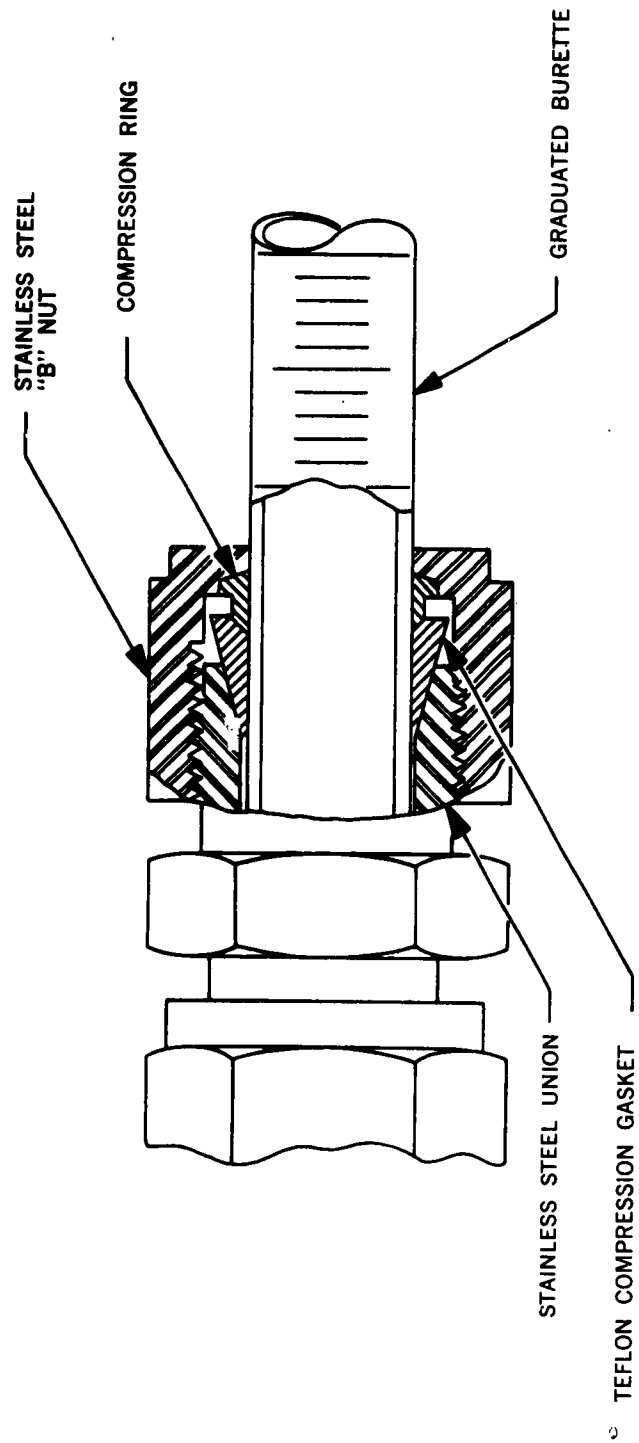


FIGURE 28

SEALING DETAIL

N<sub>2</sub>O<sub>4</sub> SPECIMEN LEAKAGE INDICATOR

### Pump Seals and Bearings

Problems were encountered with bearings and seals on both conventional and canned type pumps.

Canned Pumps - Canned pumps utilize the principle of a self-contained motor and pump to eliminate the necessity for a seal between the pump and motor. In this type of pump, the propellant serves both as a lubricant and coolant.

One pump, incorporating Graphitar-14 bearings and a stainless steel shaft, was subjected to 48 hours of continuous service with ambient temperature fuel blend and, subsequently, to a low temperature operation at  $-14^{\circ}\text{F}$ . The low temperature operation test was conducted with the pump initially stabilized at  $-14^{\circ}\text{F}$  and subsequently operated with ambient temperature fuel. Examination of the pump revealed that the pump shaft was slightly scored and the bearings were flaking at the ends, as shown in Figure 29. Subsequently, the Graphitar-14 bearing was replaced with a Graphitar-84 type and the pump performed satisfactorily for a 160-hour period in fuel blend service. A final examination revealed no evidence of shaft scoring.

A similar type pump for nitrogen tetroxide service incorporated ceramic bearings and a Stellite shaft. After 56 hours of operation, the shaft surface scored, as shown in Figure 30, causing the pump to seize. The bearing and shaft were replaced and the pump operated successfully for 160 hours in nitrogen tetroxide service.

A second type of canned pump incorporated impregnated carbon bearings of an unknown composition. This pump performed satisfactorily for 160 hours in both oxidizer and fuel blend service.

Conventional Pumps - The conventional pumps used as a part of the Wyle facility incorporated conventional dynamic seals fabricated from silver- and babbitt-impregnated carbon. A 440C stainless steel running ring formed the mating surface of the seal. These seals leaked immediately upon exposure to the fuel blend as a result of the leaching out of the silver and babbitt.

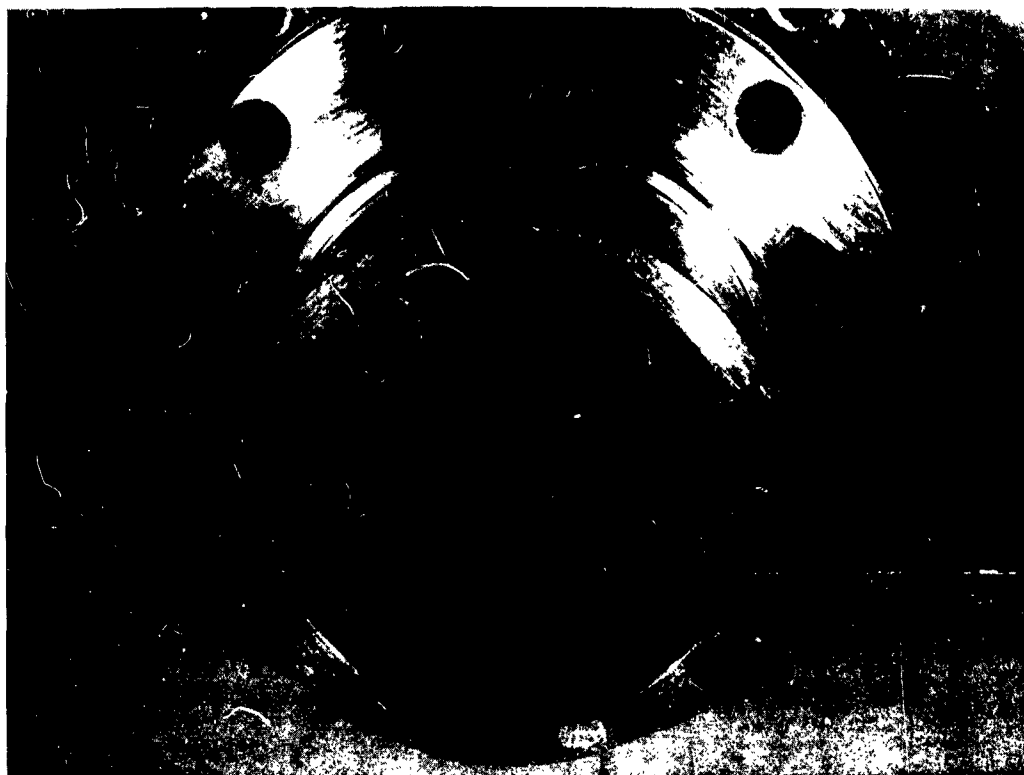


FIGURE 29

GRAPHITAR-14 PUMP BEARING

Graphitar-14 bearing in fuel pump following life cycle test. Note chipping and flaking of material from overheated bearing area.



FIGURE 30

SCORED PUMP SHAFT

$\text{N}_2\text{O}_4$  pump shaft scored as a result of possible localized overheating of ceramic bearing during life test.



The silver and babbitt-impregnated carbon seals were replaced with Graphitar-39 seals and the pump performed satisfactorily in fuel blend service over a 300-hour period. A small leak developed after 300 hours of operation, but the nominal leakage remained constant up to 500 hours of operation in fuel blend service.

A similar type pump, incorporating Graphitar-39 seals, performed satisfactorily in nitrogen tetroxide service over a 200-hour period. After 200 hours, nominal vapor leaks developed and the leakage became progressively worse until liquid was leaking after 500 hours of service.

#### Recommended Casting Procedures for Stainless Steel

Normal commercial casting sand contains iron oxides either naturally or due to previous use with cast iron. When this sand is used to cast stainless steel, the iron oxides are transferred to the stainless steel surface. If these oxides are not removed from the casting, they will eventually form rust which is incompatible with the fuel blend.

Rough castings should be acid pickled prior to machining in order to remove the surface oxides. The acid pickling should take place prior to machining, since the smooth internal surfaces of a component may be attacked by the pickling solution following machining.

Nitrogen tetroxide has, on occasion, permeated some stainless steel castings under pressure, resulting in "leakage" through the metal. It was also observed that the possibility of rust formation increases when excessive porosity exists. More stringent quality control should be effected on castings used for propellant service to prevent excessive porosity and attendant problems.

#### Paints

All of the paints and/or external coatings used on the test facility were attacked when exposed to the propellants. Difficulties were encountered in providing protective coatings for materials exposed intermittently to minor propellant spills and/or vapors.

The 6,000 gallon fuel blend storage tanks were initially coated with CAT-A-LAC epoxy resin paint. The CAT-A-LAC paint was applied over 2 inches of fiberglass insulation previously covered with a dope-coated, airplane fabric. A nominal quantity (approximately one quart) of the fuel blend was spilled on the paint surface, causing the CAT-A-LAC paint to blister. It was the vendor's opinion that the dope caused improper curing of the CAT-A-LAC.

Compatibility tests were performed on Rust Oleum No. 2764 semi-flat white, containing Rust Oleum No. 633 thinner oil, since this paint was used as a coating for the functional test cell walls. The paint was applied by dipping a stainless steel test coupon into the paint and subsequently allowing the coupon to dry over a period of 8 days. A noticeable reaction took place approximately 5 minutes after droplets of the fuel blend were placed on the coupon surface. Although the paint blistered, no combustion occurred.

The coatings described above are representative of the numerous paints and coatings that have failed to provide a protective coating on many types of vendor components, such as the pump shown in Figure 31. Until such time as paint or coating compatible with the storable propellants is developed, it is recommended that the 300 series stainless steel external surfaces be left bare.

#### Filters

The filters used and tested during the Pre-Qualification Test Program were fabricated from 304 stainless steel, using Heliarc welding. The fine mesh filter elements ranged from 30 to 70 microns absolute.

Bubble point tests conducted on the elements revealed, in many cases, that the screen contained nominal holes. These holes were apparently caused by the manufacturer's welding process which resulted in the filter end plate being brought up to a sufficiently high temperature to cause the fine mesh element to burn. One manufacturer surmounted this problem during fabrication by using nickel chrome furnace brazing when attaching the filter element to the end plate.

Normally, a nominal sized hole in a filter element can easily be repaired by brazing. However, many brazing materials are apparently incompatible with storable propellants and, for this reason, cannot be used.



FIGURE 31

TYPICAL INCOMPATIBLE PAINT APPLICATION

Paint chipping on propellant pump and mounting fixture--representative of the many applications of incompatible protective paint coatings on components and facility hardware.

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